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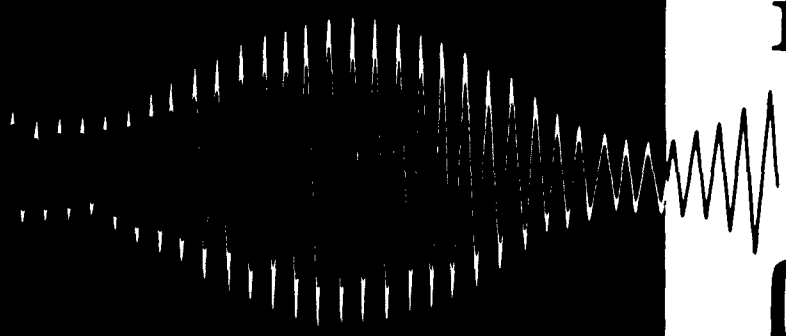
INVESTIGATION OF HIGH POWER GASEOUS ELECTRONICS

Final Progress Report

January 1, 1962 to November 15, 1962

Contract No. DA-36-039-SC-89161

U. S. Army Signal Corps
Engineering Laboratories
Fort Monmouth, N. J.



MICROWAVE ASSOCIATES, INC.

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ABSTRACT

Argon and chlorine gas cleanup and recovery in quartz vials due to the action of a microwave discharge is discussed in terms of the experimental results. The rate of gas cleanup is constant over a 3.0 Gc to 9.5 Gc frequency range. Cleanup is approximately independent of temperature over a -100°C to +600°C range whereas spontaneous thermal recovery rates are proportional to temperature. The cleanup phenomena are explained on the basis of a surface deformation-annealing sequence.

INTRODUCTION AND SUMMARY

The purpose of this program is to theoretically and experimentally investigate the nature of high power pulsed microwave plasmas in an effort to better understand and improve microwave gas switching techniques. The principal task to which we devoted our efforts was a research program directed towards understanding the nature of the gas discharge, specifically including a detailed investigation of gas cleanup.

Gas cleanup in electrodeless sealed tubes due to the action of a microwave discharge is one of the basic limitations of high power microwave gas switches. Such switches can be carefully designed to meet exacting specifications and still not be useful devices because of their short lifetimes resulting from cleanup. Though gas switches have been used for nearly two decades, this problem has only recently gained great importance with the advent of ultra high power applications. The mechanisms involved can be very complicated. In the simplest cases, as with inert gases where presumably no chemical activity is involved, cleanup is felt to be purely a mechanical process whereby gas ions are accelerated into the walls of the discharge container by the electric fields across the plasma sheath at the container surface. The ions gain sufficient energy in this process to penetrate several molecular layers of the wall surface where they become neutralized and trapped. As yet no saturation limit to this process has been found. After sufficient time has elapsed all of the gas will be driven into the container walls.

None of the gas apparently escapes the container as has been determined by careful weighings, and by the fact that containers have been heated with a naked flame resulting in 100% recovery of the initial gas fill pressure.

In the early phase of the program cleanup experiments were conducted with an argon discharge in a 7/8 inch coaxial line at 3.0 Gc and a 0.001 duty cycle. Pressure was monitored with a thermocouple gauge and tests were performed over a range of 0.1 to 45 Torr. These tests showed an initial rapid gas cleanup corresponding to the sorption of a monolayer of gas, followed by a constant cleanup rate which extended over a relatively large range of pressure. At pressures of several hundred microns the cleanup rate accelerated until the pressure dropped below a value at which an RF discharge could not be maintained. Cleanup rate was determined to be proportional to the power absorbed by the discharge and inversely proportional to average fill pressure. Incomplete outgassing of the test vial limited the sensitivity of this series of tests.

In order to eliminate experimental difficulties associated with a coaxial line discharge, a series of test were run in rectangular waveguide at 5.5 Gc and 9.5 Gc. A minimum gas volume and consequently a minimum time to cleanup all of the gas at a given initial pressure was obtained by removing the relatively large thermocouple pressure monitoring appendage. Initial pressure was measured before the quartz vial was sealed off from the vacuum system and final pressure was determined by the electrical characteristics of the discharge. The

test results indicated that the empirical formula developed from the coaxial discharge data at 3.0 Gc also approximately described cleanup of chlorine in a rectangular waveguide discharge at 5.5 Gc and 9.5 Gc and at a 0.001 duty cycle. A slower cleanup rate was obtained at a 0.25 duty cycle and the empirical formula did not fit the data. A comparison of chlorine and argon cleanup at a 0.25 duty cycle on the basis of normalized absorbed power showed an argon cleanup rate approximately three times greater than that obtained for chlorine. However, the reverse situation occurs with a chlorine cleanup rate approximately three times greater than that of argon on the basis of number of molecules cleaned up per unit time and unit discharge area. Cleanup rates obtained during this sequence of tests were only approximate values because of difficulty in thoroughly outgassing the raw quartz vials.

A further refinement of experimental technique gave more consistent cleanup data as well as providing a means for accurately monitoring spontaneous thermal re-emission of cleaned up gas. Tests were conducted at 9.5 Gc at a 0.25 duty cycle. Pressure was monitored continuously with a thermocouple. The outgassing problem was eliminated by initially baking the quartz vial for fifty hours at 900°C. These tests showed argon cleanup to be approximately independent of temperature over a -100°C to +600°C range. Cleanup was found to consist of an initial rapid cleanup rate corresponding to the sorption of a monolayer of gas, an intermediate range and a slow range in which the cleanup rate was constant over long periods of time. Spontaneous recovery of the cleaned up gas was proportional to temperature. The

recovery process consisted of an initial rapid recovery rate corresponding to the desorption of a monolayer of gas followed by a slower rate corresponding to diffusion from the interior of the walls. Assuming that cleanup rate is proportional to the number of gas atoms present yields an expression which agrees well with the experimental results.

A comparison of results obtained with the different experimental techniques shows average cleanup rates between 10^{13} and 10^{14} $\frac{\text{particles}}{\text{cm}^2\text{-min}}$. Cleanup rate is independent of frequency at a 0.001 duty cycle over a 3.0 Gc to 9.5 Gc range.

A model which explains the experimental behavior assumes "radiation damage" of the quartz lattice. Annealing of the lattice damage traps the gas ions.

Historical Survey

Siegler and Dieke[1] wrote an excellent paper on cleanup in electrodeless discharges. Their work, however, was limited mainly to pressures below 1 Torr and to inert gas fills. In order to obtain reproducible data, they found it necessary to attach an appendage with a uranium getter to purge gas impurities. Pressure was monitored by means of another appendage attached to the discharge vial. This appendage was a modified Pirani gauge consisting of a fine tungsten wire in a capillary tube. The increased gas volume due to the two appendages has the disadvantage of greatly increasing the time over which a given pressure drop will occur. In addition the modified Pirani gauge will only read pressures up to several Torr. Inert gases only can be used

because of chemical reactions with the hot wire filament.

They controlled discharge intensity by maintaining a constant light intensity as determined by a photocell. This arrangement limits the scope of the experiments. Work at this laboratory, to be described later in the report, definitely establishes a relationship between clean-up and arc loss, i.e., the power dissipated in the dicharge.

Downton[3] used the technique of adding radio active Krypton gas to inert gas fills. By monitoring radiation intensity at different locations on the discharge tube, he was able to determine pressure as a function of time. This technique also provides a good determination of just where in the walls the cleaned up gas is located. Radiation measurements as well as subsequent high temperature outgassing tests indicate cleanup is greatest adjacent to the areas of greatest discharge intensity.

Discussion of Microwave Associates Coaxial Work

Work has been performed at Microwave Associates using 5 mm OD quartz vials as the center conductor of a 7/8 inch coaxial line at 3.0 Gc. This technique offers the advantage of a uniform discharge along the outer walls of the quartz cylinder. Very high power densities can be obtained if the test unit is pressurized to prevent arcing. Water cooling must be provided to prevent melting of the center conductors and also to prevent a reduction in breakdown power caused by a decrease in gas density at high temperatures.

A schematic diagram of the waveguide test setup is shown in Figure 2. A single RF power meter is used to measure the incident power and the

power reflected and transmitted by the test section shown on Figure 1. In this manner the power absorbed by the test vial can be determined. Before each experimental run the pressure measuring thermistor is calibrated against a manometer on the vacuum system as a function of test vial fill pressure. Also the recovery time is measured as a function of fill pressure using the low power klystron source shown on Figure 2. Thus both recovery time and thermistor readings are used to check vial pressure at periodic intervals during an experimental run.

In all cases the vials were filled initially to pressures of several millimeters of mercury. The typical results obtained are shown on Figure 3(a). At first the pressure drops rapidly eventually leveling off to a nearly constant rate of decrease until a pressure somewhere in the neighborhood of one to two millimeters is reached. At this point the discharge arc loss starts to increase rapidly with decreasing pressure and the cleanup rate again increases sharply until the pressure is so low that the vial will no longer support a discharge. This sudden increase in arc loss is indicative of the catastrophic failure which results when microwave gas switches reach the end of their useable life. Usually, however, gas switches are replaced long before the onset of catastropic failure due to changes in recovery time with decreasing pressure. In a radar system using gas switches this is easily detected by changes in the character of the echo signal. The initial rapid decrease in pressure at the start of the cleanup process appears to correspond roughly with the time necessary to deposit a

monomolecular layer of gas ions on the container wall.

Great care had to be taken in all these experiments to assure that the container walls were thoroughly outgassed. In general it was found that this could not be done by conventional bakeout techniques, which were nowhere near as effective as the action of the discharge itself. The best method was found to be one of evacuating the vial and filling it with fresh pure gas after several minutes of high power operation. This process was repeated until the recovery time was found to be essentially constant over several minutes of high power operation. Figure 3(b) shows a typical result obtained when the test vials are not carefully outgassed. A pressure increase above the initial fill pressure occurs followed by a fairly rapid cleanup due to the presence of impurity gases evolved from the walls.

Another factor affecting gas cleanup is the nature of the discharge container surface. Figure 4 shows the results obtained when two successive cleanup runs are made in the same test vial. It is noticed that repeated ion bombardment apparently conditions the container walls so that successive cleanups occur at a faster rate. On the first experimental run, the power was turned off after four hours operating time and the system allowed to sit over a weekend. When the power was again turned on it was found that some of the cleaned up gas had spontaneously evolved from the container walls. In this particular experiment 18% of the original gas fill was restored in a 60 hour period. However, after a short operating time of about one hour, the pressure was reduced to below its original value of 4.75 millimeters. Furthermore it is seen that this interruption in the operating time did not

affect the overall average cleanup rate. In other words, it appears that after a certain number of hours operating time a tube will fail regardless of whether it is operated intermittently or continuously.

Careful examination of the results of several experiments have shown that gas cleanup of argon by quartz obeys the following empirical formula:

$$\left\langle \frac{dp}{dt} \right\rangle = 2.22 \times 10^{-2} \frac{\langle Pabs \rangle}{V \langle p_F \rangle} \qquad (1)$$

where $\left\langle \frac{dp}{dt} \right\rangle$ is the average cleanup rate in Torr/hour

V is the discharge container volume in cubic centimeters

$\langle Pabs \rangle$ is the average absorbed power in watts

In the above equation $\langle p_F \rangle$ is the average fill pressure in Torr given by

$$\langle p_F \rangle = \frac{p_i + p_f}{2}$$

where p_i is the initial fill pressure

p_f is the final fill pressure

Justification of Equation (1) is shown on Figure 4 where we have plotted average fill pressure in Torr against the normalized average cleanup rate in Torr per watt-hour, for five different experimental runs. The differing vial volumes were normalized by Equation (1) to a volume of two cubic centimeters. The line on this figure representing Equation (1) has been passed through the experimental point resulting from the data shown on Figure 5.

Discussion of Microwave Associates Waveguide Work Using Sealed Vials

Tests were run in waveguide mounts at 5.5 Gc and 9.5 Gc in order to eliminate water cooling, center conductor contact problems, and sputtering of the thermistor mount which occasionally arises in a coaxial system. By using the same 5.0 mm OD quartz vials, mounted in higher frequency waveguide, it was possible to obtain power densities comparable to those encountered in the 3.0 Gc coaxial work. Use of waveguide instrumentations greatly facilitated data taking in comparison to making high power measurements in 7/8 inch coaxial guide. The waveguide mount is shown in Figure 6.

It was decided to rely entirely on the discharge characteristics such as arc loss and breakdown power to monitor pressure. Figure 7 shows an average pressure calibration curve obtained from a group of vials filled at different pressures. At the conclusion of the outgassing and calibration sequence, the vial was sealed off from the vacuum system. A minimum volume to provide the quickest cleanup time is obtained with this technique. Eliminations of attached pressure monitoring devices allows the use of active gases with high electron attachment coefficients.

Results for a typical run are given in Figure 8 for a vial with a fill pressure of 1.0 Torr of chlorine. The duty cycle was 0.25. The incident power was held constant and the breakdown power and arc loss were monitored as a function of time. Arc loss as used here is defined as the power dissipated in the discharge only.

Both breakdown power and arc loss are seen to increase with time until the capsule ceased to fire at the maximum available incident power. This value of breakdown power represents a pressure of 0.2 Torr. The initial pressure was 1.0 Torr and the quantity of gas cleaned up was 0.8 Torr times the volume of .300 cc or 240 Torr-cc. An average cleanup rate is defined as

$$S = \frac{V\Delta P}{P_A \Delta t} \qquad (2)$$

where V = volume of gas in cc

ΔP = change in gas pressure in 10^{-3} Torr

P_A = arc loss in watts

Δt = elapsed time in hours

If an average value of arc loss is taken to be 22 watts, then

$$S = \frac{.300 \mid 800}{22 \mid 13.5} = .81 \frac{\text{Torr-cc}}{\text{watt hour}}$$

The discontinuity in the breakdown power curve at 6.3 hours is due to a sixteen hour hold during the test run. The breakdown power decrease probably represents a pressure increase due to gas recovery during the hold period. It is not known why the breakdown power and consequently the pressure does not recover within a short time to the values before the hold period as has been the case in previous experiments. The slope of the breakdown curve is seen to remain relatively constant before and after the hold period.

The flattening of the arc loss curve at 9.0 hours is due to an increasing time delay between the application of the RF pulse and the time at which breakdown first occurs. This has the effect of reducing the effective average incident power.

Figures 7a and 7b represent typical curves for arc loss, breakdown power and recovery time as a function of pressure for argon and chlorine gases. At very low pressures the discharge losses are dominated by diffusion of electrons to the walls. At higher pressures the discharge losses are controlled by attachment of electrons to form negative ions in the case of chlorine and by volume recombination of electrons and positive ions in the case of argon. During the course of any cleanup experiment, it is therefore inevitable that changing pressures represents significantly different discharge conditions. This leads to a rather complex situation with respect to the analysis of cleanup data. For example, in Figure 8 arc loss and probably the cleanup rate (S) is increasing rapidly with time because the discharge is in the low pressure diffusion region. On the other hand the discharge in a capsule with a

higher initial fill pressure will be in the attachment or recombination region where arc loss is less pressure sensitive. Under these conditions arc loss and cleanup rate will be approximately constant with time until the pressure is reduced to the region where diffusion becomes dominant.

A summary of life test results is presented in Tables I, II, and III. In the tables the following notation is used:

F = frequency in Gc

P_i = initial fill pressure in Torr

ΔP = change in gas pressure in Torr

V = volume of gas in cc

P_A = arc loss in watts

Δt = elapsed time to cleanup in hours

S = average cleanup rate in $\frac{\text{Torr-cc}}{\text{watt hour}}$

In addition the predicted average cleanup rate (S') and predicted elapsed time ($\Delta t'$) based on Equation (1)

$$S' = \frac{4.44 \times 10^{-2}}{P_i + .2} = \frac{V\Delta P}{P_A \Delta t'} \qquad (3)$$

are also given. Equation (1) was based on S band coaxial experiments performed at a 0.001 duty cycle with argon filled capsules.

The average cleanup rate (S) is seen to be somewhat erratic with respect to initial fill pressure. Much of this spread is believed attributable to not subjecting each capsule to an RF discharge for

complete wall outgassing before making the final gas fill.

Table I presents results obtained for chlorine gas fills at X band (9.5 Gc) and C band (5.5 Gc) for a 0.001 duty cycle. It is seen that in most cases there is agreement within a factor of two between experimental and predicted values of cleanup rate and life time.

Table II gives results for chlorine gas fills at 9.5 Gc at a 0.25 duty cycle. In this case the experimental values of cleanup rate and life time are only about one-tenth the predicted value. The inverse relationship between pressure and cleanup rate given by (3) apparently does not hold at a 0.25 duty cycle. This implies that the cleanup rate is not a simple function of arc loss. The data suggests that cleanup rate is proportional to ion energy. Higher peak powers and consequently higher ion energies are associated with low duty cycles. It is assumed that high energy ions can be trapped more readily because they will be driven deeper into the walls than low energy ions.

Table III presents the results for argon gas fills run at 0.25 duty cycle. The cleanup rates are greater than those obtained for chlorine under the same conditions but are only one-third the predicted values. The somewhat surprising result that argon cleans up at a rate which is three times faster than chlorine at a 0.25 duty cycle and on the basis of normalized absorbed power again implies that cleanup rate is not a simple function of arc loss. In this instance significantly different values of arc loss resulted in comparable life times for constant values of fill pressure and volume. However, if a comparison is made of the number of molecules cleaned up per cm^2 per minute for a given

incident power it is seen that the reverse situation holds and chlorine cleans up at a rate three times greater than that for argon.

One possible explanation for this is that the faster moving electrons create a negative wall charge. The positive ions in the argon discharge will be accelerated across the sheath towards the walls. On the other hand, the negative ions formed in a chlorine discharge by electron attachment or ionization will be repelled from the walls. Thus, the positive argon ions will bombard the walls with greater energy than the negative chlorine ions.

The assumption of a negative wall charge can be supported by an analysis of the effects of secondary electron emission by electrons.[3] The most widely investigated property of secondary emission is yield as a function of primary electron energy u_p. We introduce the symbol δ for the yield, defined as the ratio between the total number of emitted electrons and the total number of primary electrons. A typical yield curve is shown in Figure 9.

An explanation for the maximum in the yield curve can be given by defining d_p as the maximum depth of production of secondaries and d_s as the maximum depth from which secondaries can escape. Then for $u_p < (u_p)$ max., $d_p < d_s$ and $\left(\frac{\partial \delta}{\partial u_p}\right) > 0$. Similarly for $u_p > (u_p)$ max., $d_p > d_s$ and $\left(\frac{\partial \delta}{\partial u_p}\right) < 0$.

The majority of the modern yield measurements are done by an electron gun method. Here a well defined, narrow electron beam is formed and passes through a hole in the collector which surrounds the target.

The points at which $\delta=1$ will be designated at u_p' and u_p'', corresponding to positive and negative slopes, respectively, of the δ-u_p curve. When an insulator such as quartz is bombarded by a continuous flow of electrons, these points serve to separate three different types of operation. If $u_p<u_p'$, the surface charges negatively until it approaches the cathode potential. At this potential no more primary electrons can strike the surface. If $u_p''>u_p>u_p'$, the surface will charge up positively to a potential nearly equal to that of the collector, so that the space charge reduces the effective yield to unity. If $u_p>u_p''$, the surface will charge negatively until the yield is unity.

The sheath voltage of an argon microwave discharge is estimated to be 26 volts and the average electron energy is 5 electron volts. The value of u_p' for quartz is between 30 and 50.[3] Under these conditions the vast majority of plasma electrons which cross the sheath will arrive at the walls with an energy less than u_p' and a negative wall charge will be established in the steady state condition.

The situation for chlorine is considerably more complicated. A continuation of this analysis for chlorine and also for different wall materials where $\delta>1$ could provide further insight into cleanup mechanisms.

A further analysis of sheath theory will help explain additional phenomena occurring at the walls.

We have already shown that cleanup in rare gases such as argon is due to acceleration of ions into the walls where they become

neutralized and trapped. This acceleration is due to the voltage drop across the plasma sheath which forms between any discharge and the walls of its containing vessel. The reason for the formation of a sheath is that in the steady state both electrons and ions must arrive at the wall at the same rate. When a discharge is first initiated, the electrons, by virtue of their small mass, and much higher mobility diffuse to the wall first depositing thereon a layer of negative charge which forms a potential barrier so that the arrival rate of electrons is decreased to balance the ion arrival rate. One can show that for a classical Langmuir sheath, i.e., when the sheath thickness is small compared to an ion-atom mean free path, that the voltage drop across the sheath is

$$V_S = T_- \ln \sqrt{\frac{eM}{2\pi m}} \quad \text{volts} \tag{4}$$

where T_- is the electron temperature in volts

m is the electron mass

M is the positive ion mass

e is the base of the natural logarithms

For argon and $T_- = 5$ electron volts, Equation (4) predicts a voltage drop of 26 volts. This large voltage drop suggests that most of the energy of the ions striking the wall is dissipated in heat since temperatures of only a few hundred degrees are necessary to recover

all the cleaned up gas.

The ion mass flux out of the plasma is

$$\Gamma = nv = n\sqrt{\frac{2eV_s}{M}} \tag{5}$$

where $n = n_+ \approx n_-$ is the ion density in the plasma

v = ion velocity acquired in passing through the sheath

e = electronic charge

A typical ion density in our plasma is 10^{13} particles/cm^3, which by Equation (5) corresponds to a mass flux of $7\text{x}10^{20}$ particles/cm^2. When a typical cleanup rate is taken to be $7\text{x}10^{13}$ particles/min cm^2, then the sticking coefficient (ratio of number of cleaned up particles to the number of incident particles) is 10^{-7}. Thus, if the mechanism postulated above is a correct one then cleanup is seen to be a relatively rare occurrence since only one ion out of every ten million arriving at the container walls is trapped.

An estimate of the bombarding energy of the ions can be obtained by assuming a threshold value for cleanup. Only those incident ions with an energy greater than the threshold value will be trapped and cleaned up. Since only 1 out of 10^7 incident ions is trapped, the ions which are cleaned up can be considered to represent those ions in the high energy tail of a Maxwellian distribution with an average value corresponding to the voltage drop across the sheath. Under these

conditions the minimum ion energy at the threshold level is 350 electron volts.

Discussion of Waveguide Work with a Continuous Pressure Monitor

Because of the wide variation in cleanup rates obtained from the sealed vials, the experimental procedures were charged to give more consistent cleanup results and to provide means for measuring the spontaneous recovery process. Pressure was continuously monitored in a manner similar to that used earlier in the 3.0 Gc coaxial work. A discharge was produced at 9500 Mc in a quartz vial of 5 mm outside diameter by 1 mm wall. The vial was shunt mounted as shown in Figure 1a and was connected to a vacuum system as shown in Figure 6. The microwave waveguide system is also shown in Figure 10.

Power from the transmitter passes through the ferrite circulator to the waveguide arm containing the quartz vial. Nearly all of the power is reflected by the quartz vial discharge back to the circulator. The reflected power passes through the circulator again and on to load A where it is dissipated. A waveguide short is placed 90° behind the capsule to lower the breakdown power by placing a voltage maximum at the quartz vial.

A thermistor connected to a bridge circuit was used to monitor pressure. During a test run the vial was isolated from the main vacuum system by means of valve V_1. Vacuums of the order of 10^{-7} Torr were obtained prior to gas filling.

Ambient temperature was controlled by the small oven shown in Figure 10. Vial temperature was monitored by means of a thermocouple embedded in the waveguide mount as shown in Figure 6. All pressure readings were adjusted to assure that pressure variations are the result only of changes in the number of molecules present.

An initial bakeout was performed for eighteen hours at 500°C. However, when a pressure of 1000 microns of argon was admitted to the vial and a 400 watt average power discharge at a 0.25 duty cycle was initiated at 25°C, it was found that the pressure continued to rise steadily for about a 300 micron total increase over a ten hour period. This indicated that the discharge was liberating gas impurities from the wall at a rate greater than the argon cleanup rate and that improved outgassing techniques were required.

The vial was again pumped to a vacuum of 10^{-7} Torr. Valve V_1 was closed to isolate the main vacuum system and the temperature raised above 500°C. The amount of thermal outgassing which occurred is shown in Figure 11. The pressure rose from zero to 600 microns after eight minutes at 600°C. A rapid increase in pressure to 2700 microns occurred in the next twenty minutes when the temperature was increased to 800°C. This was followed by a slower rate of gas evolution to 2900 microns in the following sixty minute interval. Another rapid increase in gas evolution occurred with a subsequent temperature increase to

1000°C. After a total time lapse of 300 minutes the pressure in the vial had risen to 4500 microns.

A comparison can be made between discharge outgassing and thermal outgassing by multiplying the thermal outgassing pressure readings by the ratio of quartz surface area in the oven to the discharge surface area. This value is approximately 0.1. Under these conditions the 300 micron pressure increase due to discharge outgassing is comparable to an adjusted 300 micron thermal outgassing which occurs at 800°C. Accordingly, the severe outgassing action of a high power microwave discharge requires extremely high initial bakeout temperatures to obtain reproducible results. A continuation of the bakeout for fifty hours under vacuum at 900°C resulted in a system hold-off of less than 0.6 microns per hour at 900°C.

These results strongly indicate that the wide variations in cleanup rates obtained earlier are the result of incomplete outgassing. Vials with unusually slow cleanup rates undoubtedly contained large quantities of residual gases trapped in the walls and which are released by the discharge. These residual gases could have the effect of increasing the initial fill pressure many times with a corresponding decrease in the apparent cleanup rate.

Typical cleanup-recovery isotherms are given in Figures 12, 13, and 14. Three cleanup-recovery cycles at 200°C are shown in Figure 12.

Initially a rapid cleanup rate is observed which starts at point A. This is followed by a slower and approximately linear rate beginning at point B. At point C the discharge was terminated and spontaneous re-emission of the cleaned up gas occurs. The discharge was re-established at point D and cleanup continued to point E. A second recovery period occurred between E and F followed by a third cleanup-recovery cycle between points F, G, and H.

The three recovery periods are nearly identical as evidenced by a 65 micron pressure increase during the first sixty minutes of each recovery portion of the cycle. The cleanup portions of the cycle are similar in shape but do not repeat exactly. This somewhat erratic behavior during cleanup is typical of all the tests when short time intervals are examined.

At point H the temperature was raised to 500°C and full recovery of the cleaned up gas was obtained at point J. Tests at other ambient temperatures indicated that a comparable 300°C increase above the temperature at which cleanup took place was necessary to effect full recovery.

A 400°C cleanup-recovery curve is shown in Figure 13. In this figure, the additional data points show more clearly the erratic nature of the cleanup process. The constantly changing cleanup rate precludes the establishment of a reproducible instantaneous cleanup rate. It appears that time intervals of the order of ten hours are necessary to determine a reproducible average cleanup rate.

It can be seen that the recovery portion of the curve consists

of two sections. A rapid initial recovery rate occurs between points B and C in Figure 13. This is followed by a slower rate of gas evolution between points C and D. The data in Figure 13 is redrawn in Figure 14 on a compressed time scale. Additional recovery data has been added. Gas is still being evolved twenty-two hours after the discharge has terminated although at an ever decreasing rate. It appears that gas evolution at 400°C will cease at some value short of full recovery.

A series of recovery isotherms showing the temperature dependence of the recovery process are given in Figure 15. In the figure the number of atoms desorbed per cm^2 of discharge area are plotted as a function of time. The recovery rate increases with temperature from a negligible value at 25°C. The elapsed time before the transition occurs between the initial rapid recovery and the slower long term recovery is seen to decrease with increasing temperature.

Figure 16 shows a series of cleanup or sorption isotherms on an expanded time scale for the first twenty minutes of cleanup. The number of atoms sorbed per cm^2 of discharge area are plotted against time. A very rapid cleanup during the first few minutes is followed by a constant cleanup rate which is approximately independent of temperature from -100°C to +600°C. It is noted that the curves follow no particular temperature sequence.

The large variations between curves during the first few minutes is believed to be due to residual gas atoms on the surface prior to the initiation of a discharge. The cleanest surface would be present at 600°C because few impurity atoms could be adsorbed at

such a high temperature. Any impurities present would be adsorbed outside of the oven area at lower temperatures. At the other extreme the added complication of cryogenic pumping is present. The -100°C temperatures were obtained by pouring liquid nitrogen into the oven volume. The discharge area was then at the lowest temperature in the cleanup system. Impurity gases could then be preferentially adsorbed in the discharge area. A refinement of the experimental technique used would be the addition of a second nitrogen cold trap in the cleanup system in addition to the one already present in the main vacuum system. The new cold trap would be at -195°C because of an intimate contact with the liquid nitrogen. This would provide an impurity adsorption area at least 95°C cooler than the discharge area which would not be disturbed by the discharge. It is quite possible that such carefully controlled vacuum techniques would also show that cleanup is independent of temperature even during the first few minutes that the discharge is on.

The cleanup data in Figure 16 is extended to longer time intervals in Figure 17. A transition occurs at about 20 minutes to a second slower constant cleanup rate which is also approximately independent of temperature.

All of the reported data in Figures 2 through 8 was obtained from a single quartz vial. At the conclusion of the tests the vial was opened and examined with a 25X microscope. No signs of sputtering or disordering on the interior surface was evident. In fact it appeared that the interior surface was smoother than was the case before testing.

The outside of the vial showed signs of devitrification on the side opposite the discharge.

DISCUSSION

An understanding of the experimental result requires an explanation for the following observed phenomena:

1. Cleanup is independent of ambient temperature.
2. Cleanup proceeds in an irregular manner over short time intervals.
3. Saturation has never been observed in an electrodeless discharge with the result that many hundreds of equivalent monomolecular layers of gas are cleaned up.
4. Spontaneous emission and full recovery of cleaned up gas occurs at temperatures which are substantially lower than that required for complete outgassing of quartz samples not previously exposed to an electrodeless discharge.

A model which qualitatively expalins the observed results assumes a deformation and erosion of the surface due to the action of the discharge or "radiation damage" followed by an annealing action. Baker[4] assumes a similar process to explain "secondary" gas cleanup in an ionization gauge.

When positive ions are shot into the walls of the quartz, they must lose their energy of translation in order to attain thermal equilibrium with the solid. This energy is quite likely to be lost in creating point defect lattice damage or disorder in the surface of the solid.

A short time later the surface anneals and the gas atoms become trapped in the lattice structure.

Such a process is consistent with the cleanup temperature independence. The deformation of the surface is primarily a mechanical process. The random thermal energy acquired through temperature changes is small compared to the energy acquired from ionization of the gas atom.

If the cleanup were similar to ordinary adsorption a temperature dependence of the following form could be expected[e]

$$\frac{dn}{dt} = \frac{p}{\sqrt{2\pi mkT}} \sigma f(\theta) \exp - \left(\frac{E_{act}}{RT}\right) \qquad (6)$$

Here $\frac{p}{\sqrt{2\pi mkT}}$ is the number of molecules arriving per cm^2 and per second

$\exp(-E_{act}/RT)$ is the fraction of molecules possessing the energy E_{act} or higher

$f(\theta)$ is the chance that a molecule hits a site which is available for adsorption

σ is the chance that a molecule with sufficient energy which hits an available site is adsorbed.

Hence, ordinary adsorption theory is not applicable. It is felt that a comparable expression can probably be developed for the cleanup case if ion energies are substituted for kinetic energy of the molecules

(kT) in the above expression.

The deformation and annealing process is also consistent with the irregular nature of cleanup. Any discontinuities and voids in the quartz lattice could be expected to influence cleanup in an erratic manner.

The absence of saturation and the sorption of hundreds of equivalent monomolecular layers of gas could be due to an unending erosion and deformation process in which a honeycomb type surface structure is formed. The erosion process can also create a large increase in surface area with respect to an initially smooth surface.

It is instructive at this point to compare the initial cleanup with the number of equivalent monomolecular layers of sorbed argon. Taking the diameter of the argon atom[5] equal to 3×10^{-8} cm and assuming a smooth surface yields a value of 0.14×10^{16} molecules per equivalent layer. Figure 15 gives a value of 2.6×10^{16} molecules/cm^2 sorbed during the first five seconds of the discharge at 600°C. This initial sorption represents the addition of 18.5 equivalent monomolecular layers to a smooth surface. An alternate viewpoint is to consider the initial rapid sorption is due to the addition of a single monomolecular layer and that the discharge has created a surface roughening factor of 18.5 which has increased the surface area 18.5 times that of a smooth surface.

The value of 2.6×10^{16} molecules/cm^2 sorbed during the first five seconds of a 600° discharge is the result of a somewhat difficult measurement because of the dynamical character of the event. With this

condition in mind, it is possible to interpret the number of molecules in an equivalent layer as being 1.3×10^{16}. This is also the number of molecules desorbed in Figure 15 at the transition between the initial rapid desorption and later slow desorption during the recovery period. The transition point occurs at a constant value of 1.3×10^{16} molecules/cm^2 for temperatures between 200 and 600°C.

Thus the extremely rapid cleanup during the first few seconds of the discharge represents the sorption of an equivalent monomolecular layer of argon and the relatively rapid initial recovery after the discharge is terminated represents the desorption of an equivalent monomolecular layer. The slower recovery at longer time intervals can be attributed to the longer times required for gas to diffuse from beneath the surface. The value of 1.3×10^{16} molecules/cm^2 obtained from Figure 15 probably represents a more accurate value of the number of molecules in an equivalent layer. In this case the increase in surface area is 9.25 relative to a smooth surface.

The difference between the temperature required for complete initial outgassing and that required for full recovery of cleaned up gas can also be explained by the surface deformation model. It can be assumed that untreated quartz will contain gas trapped throughout the volume at various levels of binding energy. Baking under vacuum for long periods of time at 500°C, for example, will release a portion of the trapped gas by diffusion from the interior. The fraction of gas released at a given temperature will depend on the energy of activation of diffusion.

If a discharge is initiated at a partially outgassed wall, the wall erosion caused by the discharge can expose trapping sites whose energy of activation for diffusion is greater than the corresponding bakeout temperature to which the wall had previously been exposed. The energy of activation for surface desorption is normally less than that for diffusion and the newly exposed trapped gas is readily desorbed.

It appears that the binding energy of the argon atoms sorbed by the cleanup mechanism is less than that of the impurity gases originally present in the quartz. It was mentioned earlier in the report that the temperature required for full recovery increases with increasing cleanup temperature. Consequently, binding energy and the energy of activation for diffusion from beneath the surface increase with increasing cleanup temperature.

An alternate explanation of the cleanup phenomena is the direct penetration and entrapment of ions in the interior of the walls as opposed to a surface entrapment and annealing sequence. This alternate explanation is not considered to be a likely process, however, for the following reasons:

1. Blodgett and Vanderslice[7] have shown in the case of a DC discharge with metal electrodes, that ions with energies of several hundred electron volts do not penetrate the electrode surface. Cleanup under their experimental conditions occurred by the burial of ions with sputtered electrode material. It is estimated that the average ion energy in the electrodeless discharge under discussion is about 500 electron volts.

2. The work of Nielson[8] has shown that the depth of penetration in metals of inert gas ions with energies of 50,000 electron volts is about 200 molecular layers. Such high ion energies are two orders of magnitude greater than in the electrodeless discharge while the depth of penetration is less than that calculated for the electrodeless discharge case.
3. Norton and Tucker[9] have recovered at 400°C inert gases which were cleaned up during a 40,000 electron volt bombardment of uranium. When krypton is produced in uranium through fission, much higher temperatures are required for release of the trapped gas[10,11] One essential difference in the two cases is the mode of introduction of the gas. Ion bombardment is from outside into one side only, and it is possible that impact of these energetic ions produces damage paths in the metal through which the gas can eventually emerge. For fission products such damage paths would be produced in the interior and in general not lead to the surface.

Thus, even in the case where sufficient ion energy is available for deep penetrations, the relatively low recovery temperature indicates that entrapment still occurs close to some interior surface which has access to the exterior surface of the material.

The previous discussion has shown the existence of three distinct cleanup regions: (1) an initial rapid cleanup where an equivalent monomolecular layer of gas is deposited in a few seconds, (2) an intermediate region where a fraction of an equivalent monolayer is

deposited during the next twenty minutes, and (3) a slower linear region after approximately twenty minutes where the surface deformation annealing sequence occurs.

It is reasonable to assume that cleanup is proportional to the density of molecules in the gas phase. In the first two regions there appears to be a finite number of available trapping sites. In order to express this mathematically, we chose time varying cleanup coefficients with characteristic half lives. In the third region the cleanup coefficient is independent of time in the regions of present experimental interest. These coefficients and characteristic half lives can be determined experimentally. Thus cleanup can be expressed as

$$\frac{dN}{dt} = -(\alpha_1 e^{-\frac{t}{\tau_1}} + \alpha_2 e^{-\frac{t}{\tau_2}} + \alpha_3)N \qquad (7)$$

where N = molecular density

t = time

α_1, α_2, α_3 = cleanup coefficients

τ_1 = initial cleanup characteristic half life

τ_2 = intermediate cleanup characteristic half life

Equation (7) is derived in an attempt to find an empirical formula to describe our results as a first step to finding a proper physical explanation.

The independence of cleanup on temperature indicates that outgassing rates during cleanup are negligible compared to cleanup rates. Accordingly, it is not necessary to add any outgassing terms to (7).

A further simplifying assumption considers the molecular density to be constant. During the course of the present experimental work, molecular density did not drop more than 30% below its initial value (N_o). Under these conditions of putting $N = N_o$ on the right hand side of (7), the solution is

$$N = N_o \left[1-\alpha_1\tau_1(1-e^{-\frac{t}{\tau_1}}) + \alpha_2\tau_2(1-e^{-\frac{t}{\tau_2}})-\alpha_3 t\right] \qquad (8)$$

Equation (8) is plotted in Figure 18. It is seen that an excellent agreement can be obtained between the empirical theory and experiment with appropriate values of the parameters. It is essentially a curve fitting procedure but it will be helpful in the investigation of the cleanup process.

Although (8) appears to exhibit a linear decay after $t = \tau$ where τ is sufficiently large to make the first two terms much smaller than the third term, it is probable that the third term can be represented by a slow exponential decay. In the region $t>\tau$ we may write for Equation (7):

$$\frac{dn}{dt} = -\alpha_3 N \tag{9}$$

without making the simplifying assumption of constant molecular density. The solution of (9) is

$$N = N_{o\tau}\, e^{-\alpha_3 (t-\tau)} \tag{10}$$

where $N = N_{o\tau}$ at $t = \tau$.

Equation (10) represents a slow decay to zero molecular density. The decay will appear to be linear for an initial small range where t is small. This simple law can not be extended over large pressure changes because changing discharge conditions will introduce additional considerations resulting in changes in the value of α_3. An examination of how the various plasma parameters vary with pressure will be the subject of future investigations.

CONCLUSIONS

Preliminary outgassing of quartz vials at 900°C is necessary to obtain reproducible cleanup data in an electrodeless discharge. Argon cleanup is approximately independent of temperature over a -100°C to +600°C range. Three distinct cleanup regions occur:

1. An initial rapid cleanup during the first few seconds corresponding to the sorption of a monomolecular layer of gas.
2. An intermediate cleanup rate during the first twenty minutes of the discharge.
3. A slower cleanup rate region after the first twenty minutes in which cleanup proceeds at a linear rate.

Assuming that cleanup rate is proportional to the number of gas atoms present yields an expression which agrees well with the experimental results.

Spontaneous recovery of cleaned up gas is proportional to temperature. Recovery can be divided into two regions:

1. An initial rapid recovery period corresponding to the desorption of a monomolecular layer of gas.
2. A slow recovery rate period corresponding to the diffusion of gas from layers beneath the surface.

Full recovery of cleaned up gas can be achieved by heating to temperatures about 300°C greater than the temperature at which cleanup occurred.

Cleanup rate is independent of frequency at a 0.001 duty cycle over a 3.0 to 9.5 Gc range for both coaxial and rectangular waveguide geometries. Average cleanup rates varied between 10^{13} and 10^{14} particles/cm^2 min. The sticking coefficient or the ratio of cleaned up particles to the number of incident particles is 10^{-7} indicating that cleanup is a relatively rare occurrence. The minimum ion energy for argon cleanup is estimated to be 350 volts.

A model which explains the cleanup phenomena after the initial rapid sorption of a monolayer of gas assumes point defect lattice damage at the wall surface. A later annealing sequence causes the sorbed atom to become trapped in the wall lattice. Continued erosion at the surface creates pits and craters which may be many hundreds of equivalent molecular layers deep. Subsequent annealing produces a honeycomb structure entrapping the cleaned up gas within the walls of the discharge container.

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TABLE I

Cleanup Life Test Results for Chlorine at 0.001 Duty Cycle

F	P_i	P_A	V	Δt	S	S'	$\Delta t'$
Gc	Torr	Watts	cc	Hours	$\frac{\text{Torr-cc}}{\text{Watt-Hour}}$	$\frac{\text{Torr-cc}}{\text{Watt-Hour}}$	Hours
5.5	15	31	4.650	182	12.22×10^{-3}	2.96×10^{-3}	412
	30	31	4.650	1000+	298	1.47	4000
	30	31	4.650	2000+	298	1.47	4000
9.5	15	15	2.020	570	3.50	2.96	673
	15	42	2.020	89	8.12	2.96	245
	20	18	2.430	1500	1.78	2.22	1210

TABLE II

Cleanup Life Test Results for Chlorine at 0.25 Duty Cycle

F	P_i	P_A	V	Δt	S	S'	$\Delta t'$
Gc	Torr	watts	cc	Hours	$\frac{\text{Torr-cc}}{\text{Watt-Hour}}$	$\frac{\text{Torr-cc}}{\text{Watt-Hour}}$	Hours
9.5	.5	16	.298	2.7	2.03×10^{-3}	63.5×10^{-3}	.09
	.5	16	.285	2.5	2.14	63.5	.08
	1.0	16	.313	7.0	2.23	37.0	.42
	1.0	22	.300	13.5	.81	37.0	.30
	1.5	19	.335	49	.47	26.1	.89
	1.5	9	.300	150	.29	26.1	.89
	2.0	16	.210	11.5	2.05	20.2	.90
	4.0	7	.200	122	.715	10.6	10.2
	4.0	7	.200	157	.692	10.6	10.2
	6.0	7	.212	192	.915	7.17	24.5
	8.0	35	.360	128	.630	5.42	14.8
	8.0	35	.360	152	.537	5.42	15.0
	8.0	35	.280	48	1.62	5.42	14.3
	8.0	35	.280	72	1.08	5.42	14.3
	8.0	50	.580	300+	.822	5.42	14.3
	9.0	7	.200	400+	.640	4.88	45.5

TABLE III

Cleanup Life Test Results for Argon at 0.25 Duty Cycle

F	P_i	P_A	V	Δt	S	S'	$\Delta t'$
Gc	Torr	watts	cc	Hours	$\frac{\text{Torr-cc}}{\text{Watt-Hour}}$	$\frac{\text{Torr-cc}}{\text{Watt-Hour}}$	Hours
9.5	2.0	2.5	.195	24	5.83×10^{-3}	20.2×10^{-3}	6.9
	2.0	2.5	.190	32	4.27	20.2	6.9
	4.0	2.5	.196	135	2.21	10.6	28.1
	4.0	2.5	.198	71	4.23	10.6	28.1
	6.0	2.5	.195	192	2.36	7.17	63.3
	9.0	2.5	.200	400+	1.80	4.88	148
	9.0	2.5	.200	400+	1.80	4.88	148

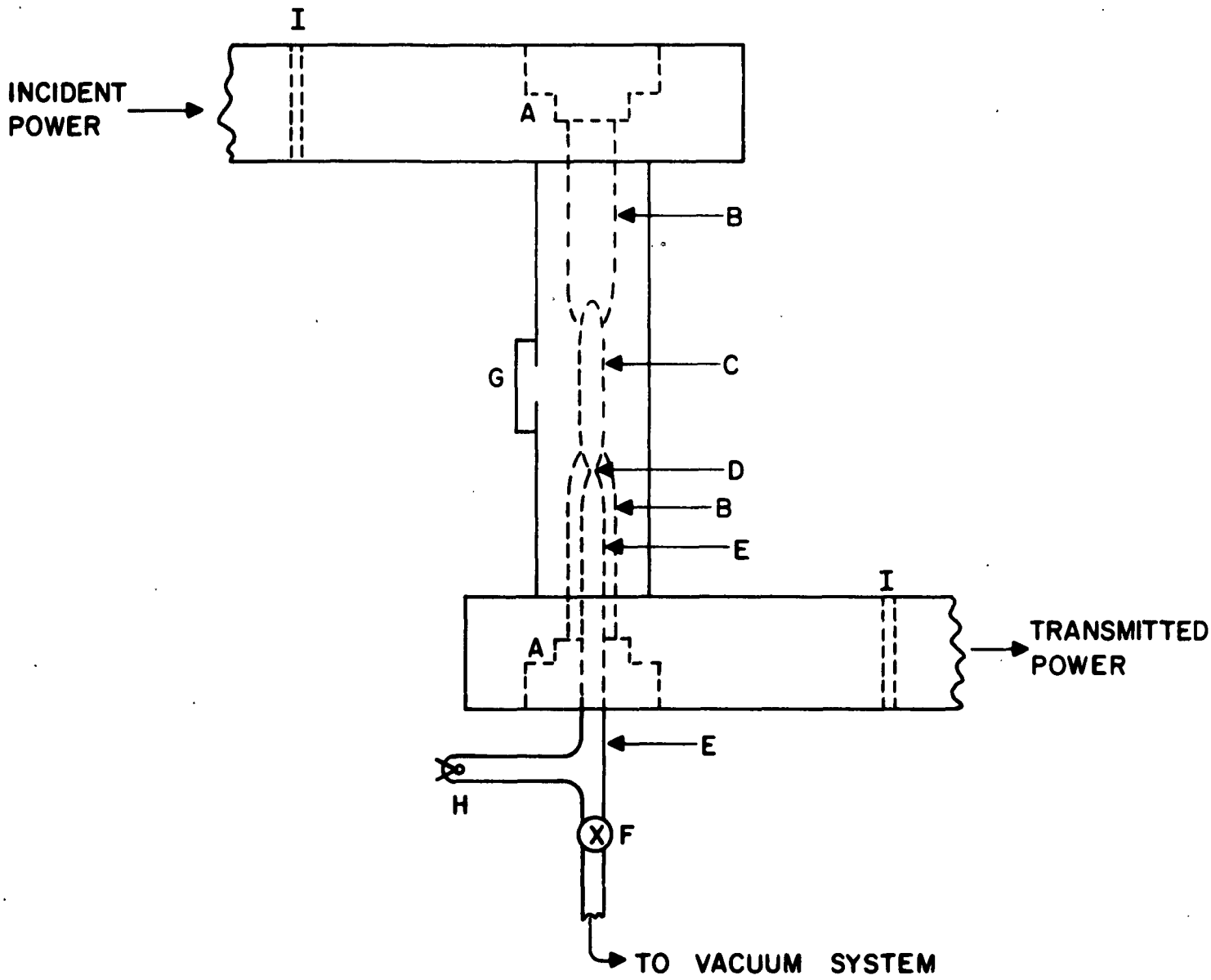


FIGURE I

COAXIAL WAVEGUIDE APPARATUS
FOR GAS CLEANUP STUDIES

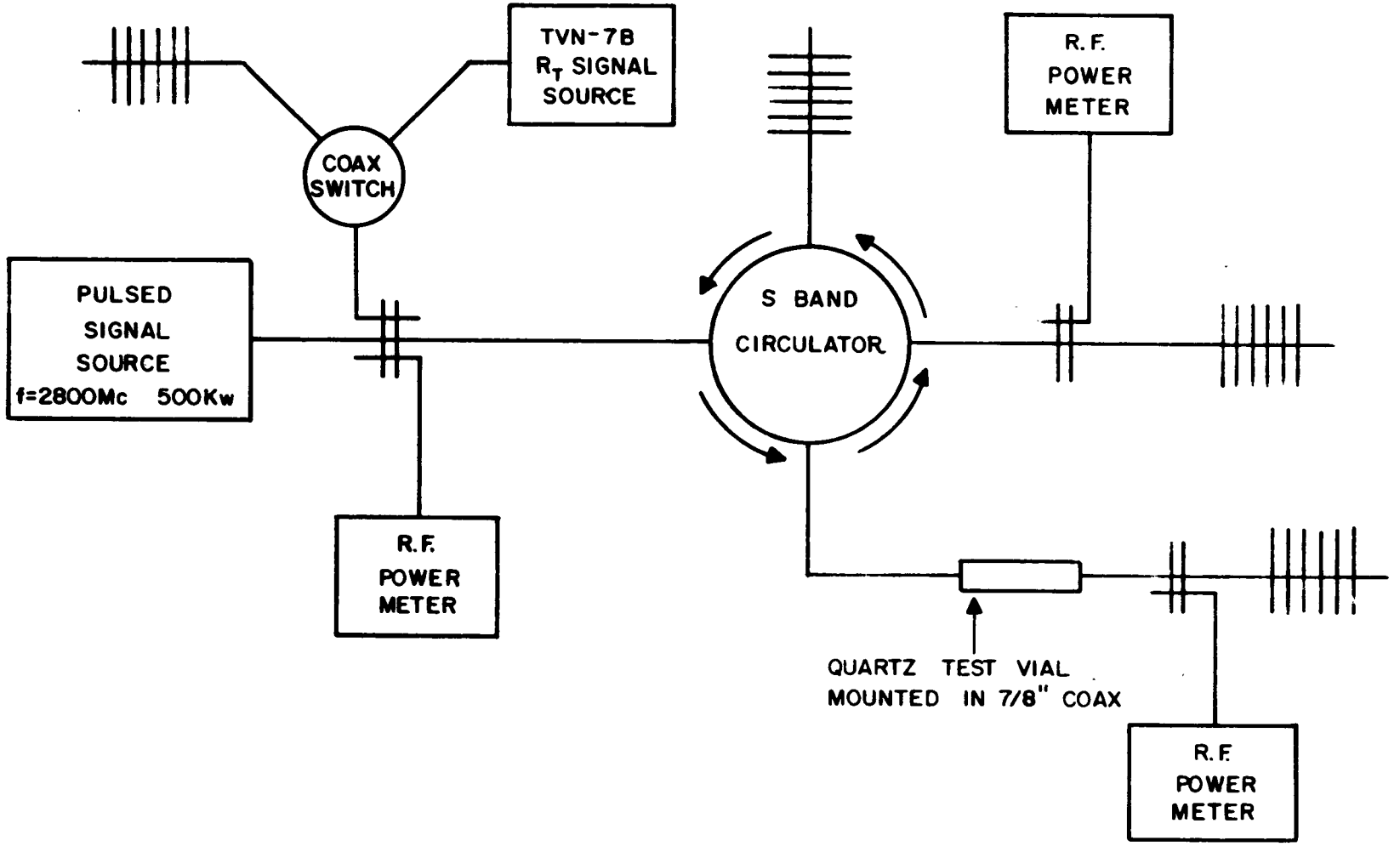
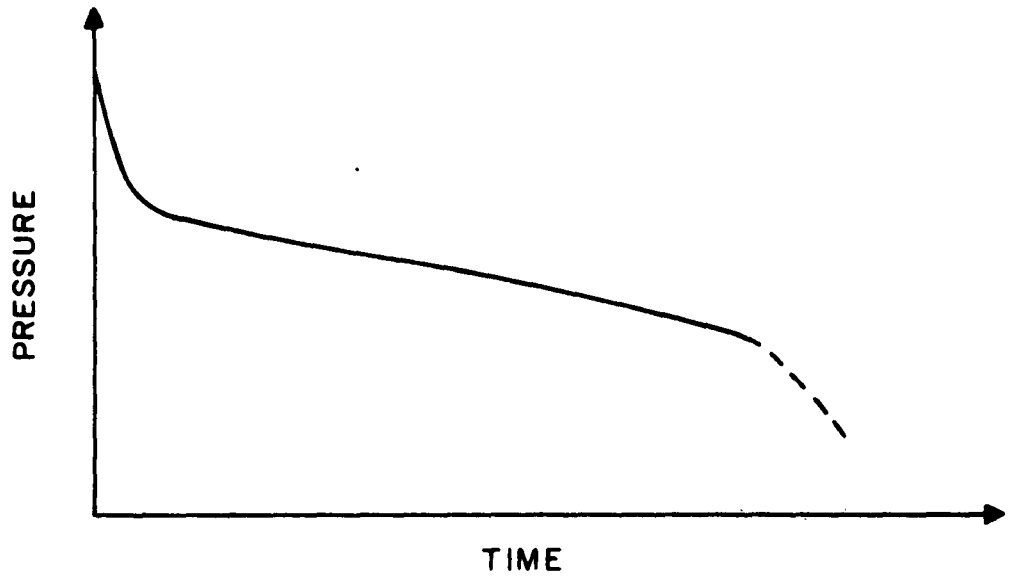
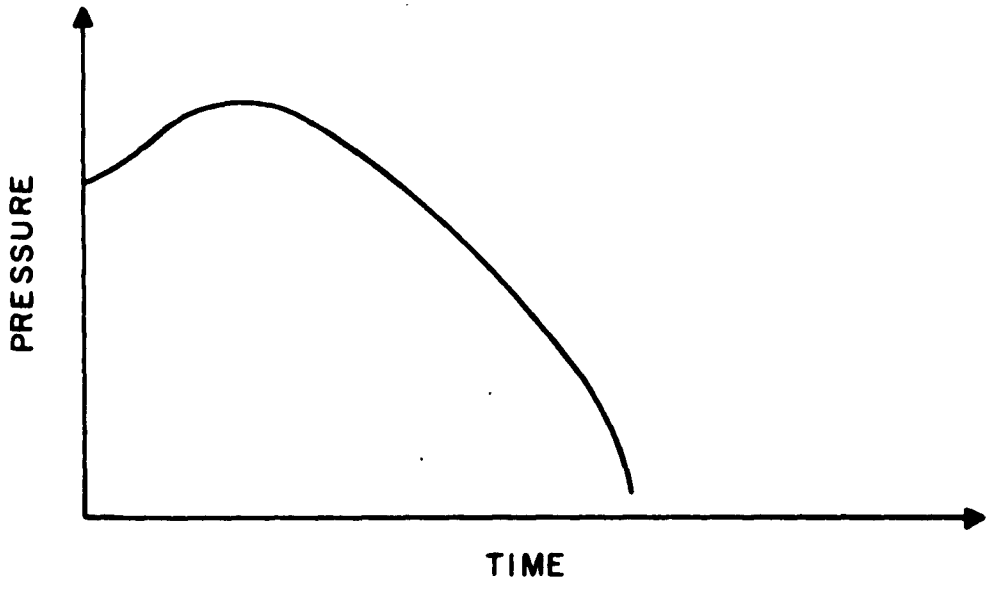


FIGURE 2

EXPERIMENTAL SETUP FOR MEASURING GAS CLEANUP



3 (a.) RESULTS WITH PURE GAS FILL



3(b.) RESULTS DUE TO OUTGASING OF WALLS

FIGURE 3
TYPICAL RESULTS OF CLEANUP STUDIES IN ARGON

FIGURE 4

AVERAGE FILL PRESSURE VERSUS AVERAGE CLEANUP RATE

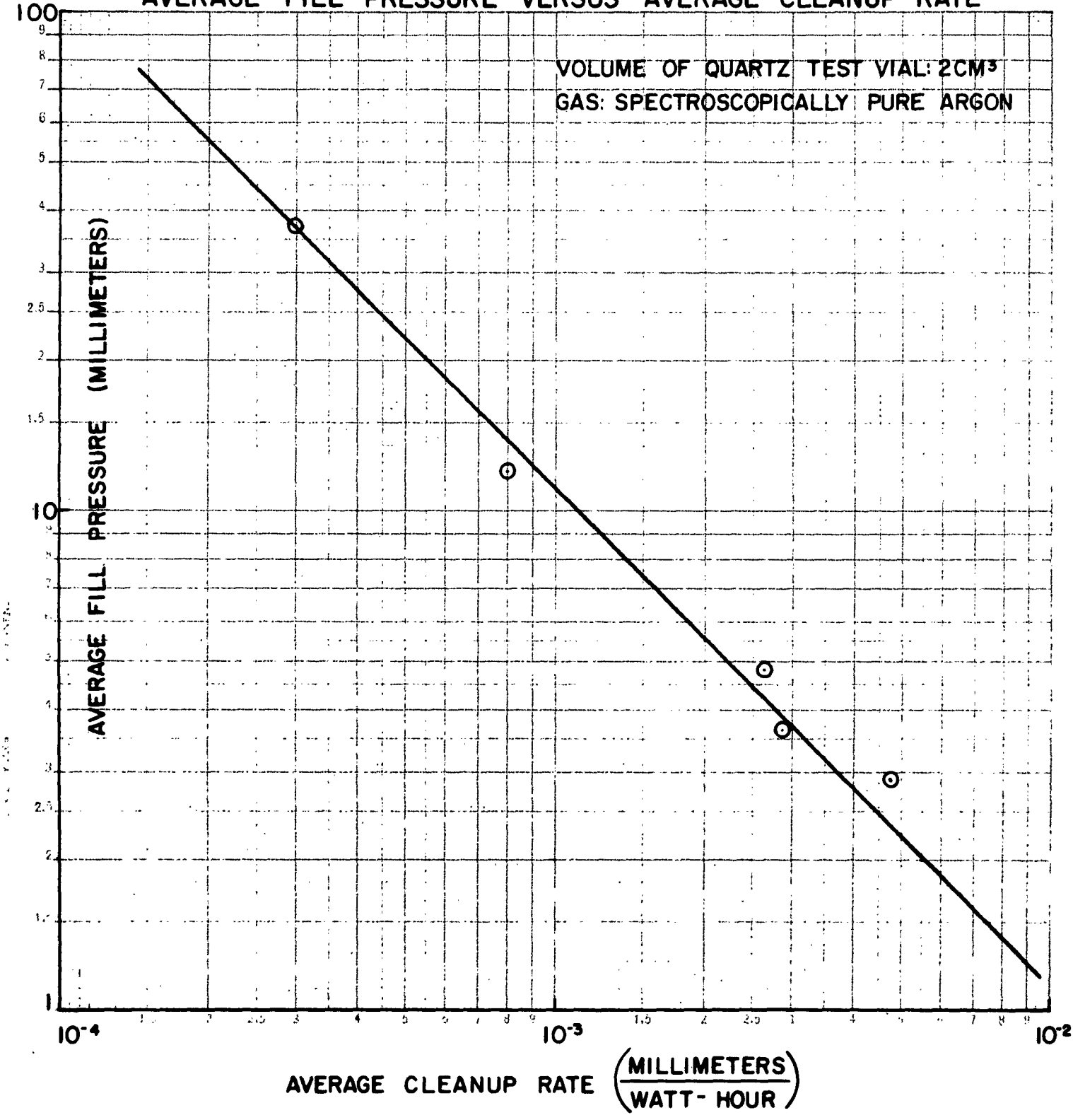
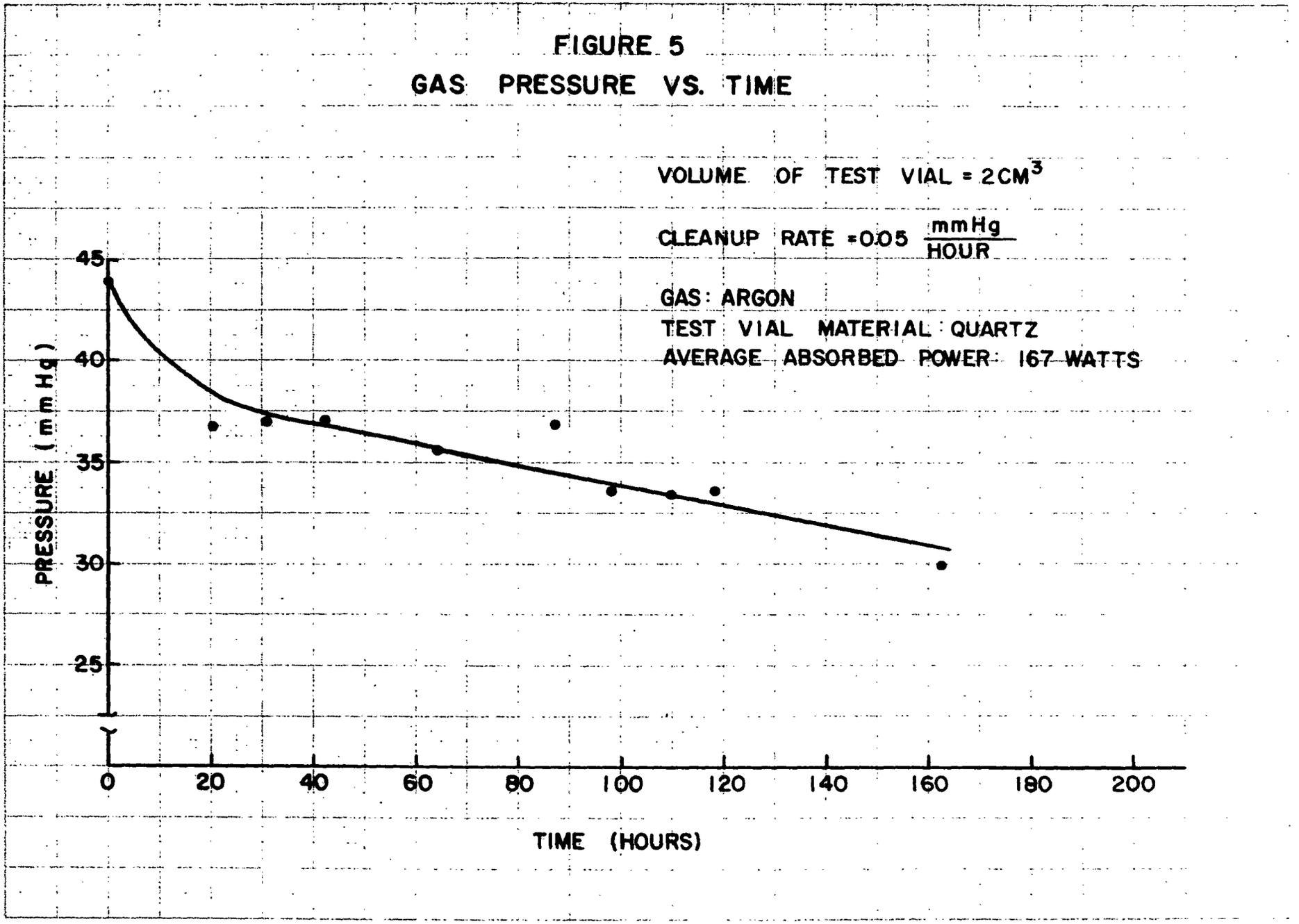


FIGURE 5
GAS PRESSURE VS. TIME



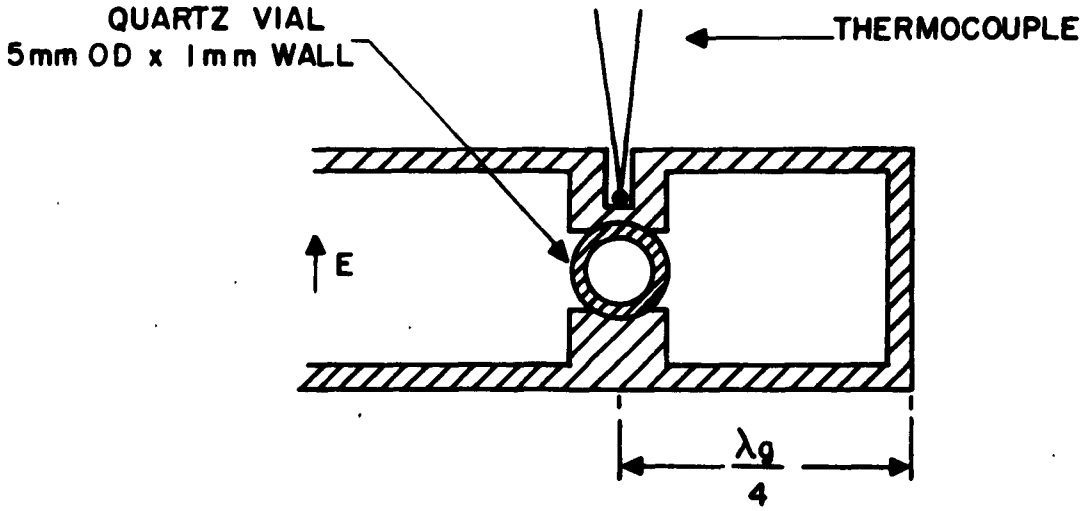


FIGURE 6

QUARTZ VIAL
WAVEGUIDE CROSS SECTION

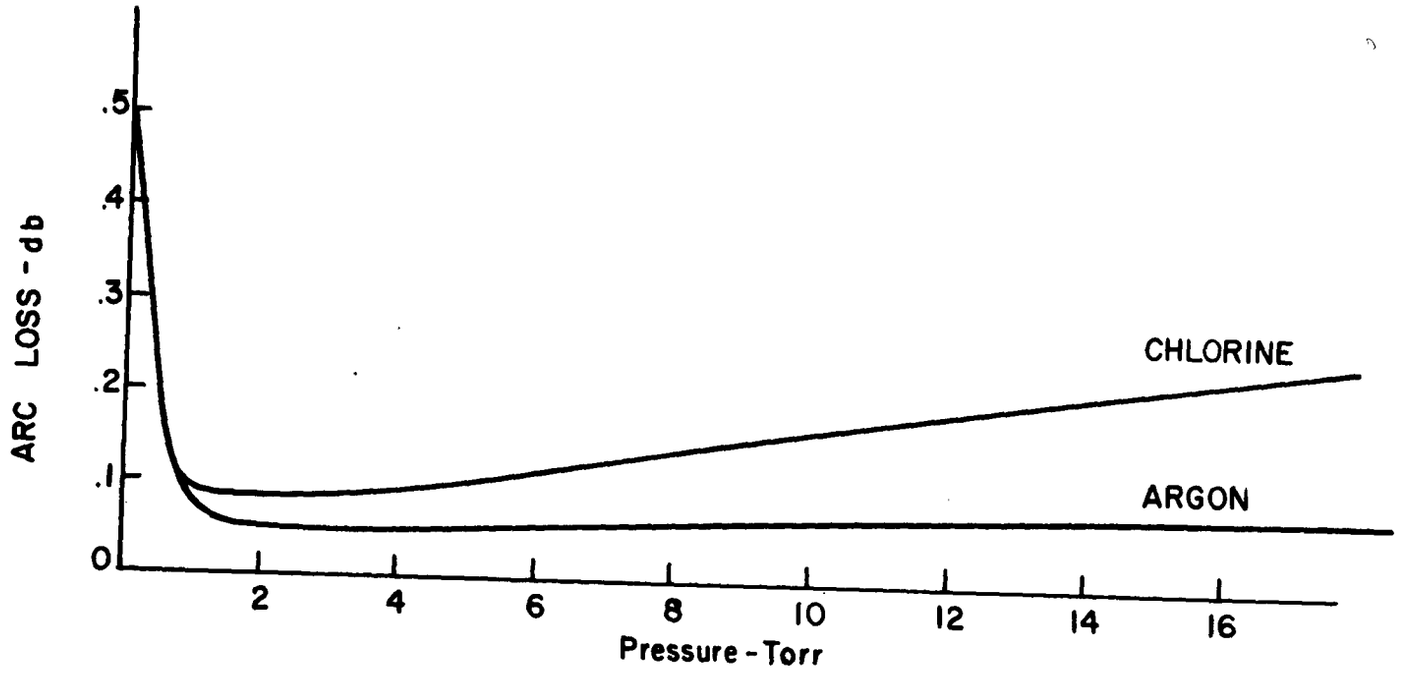


FIGURE 7A
ARC LOSS VS PRESSURE

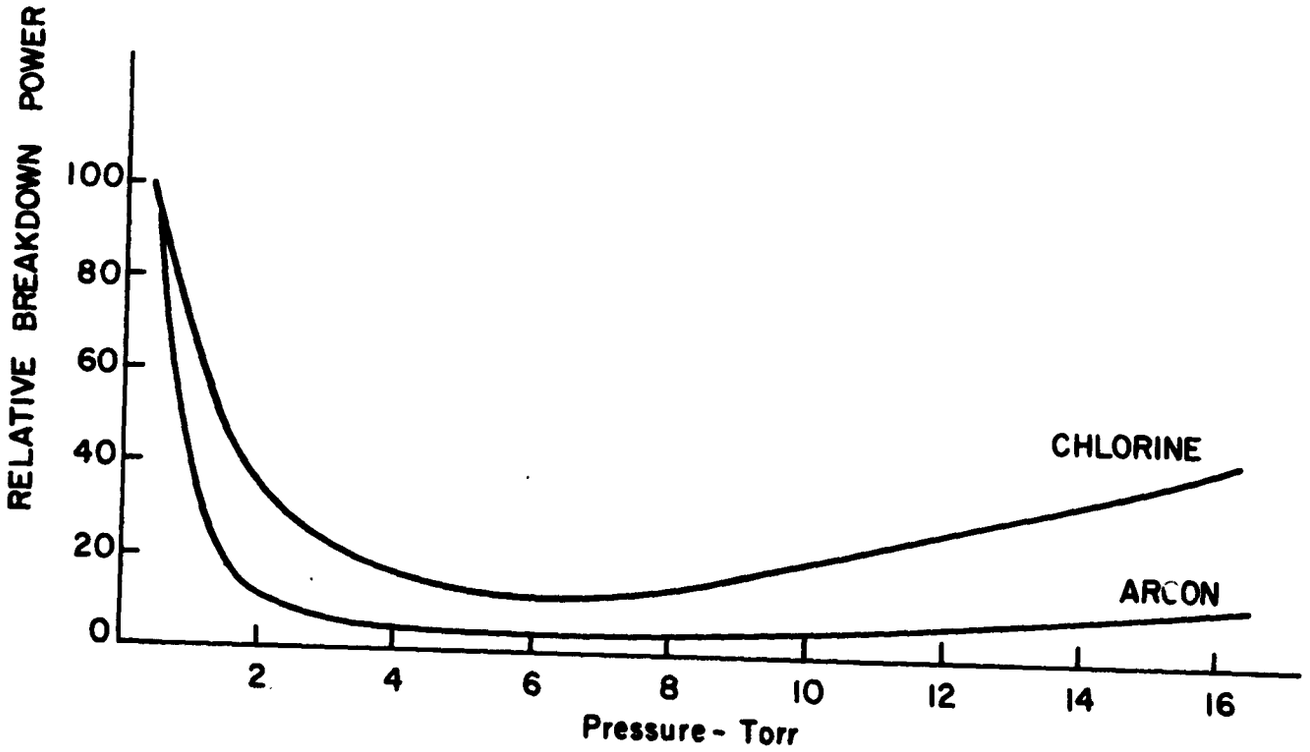


FIGURE 7B
BREAKDOWN POWER VS PRESSURE

FIGURE 8

BREAKDOWN POWER AND ARC LOSS VS TIME

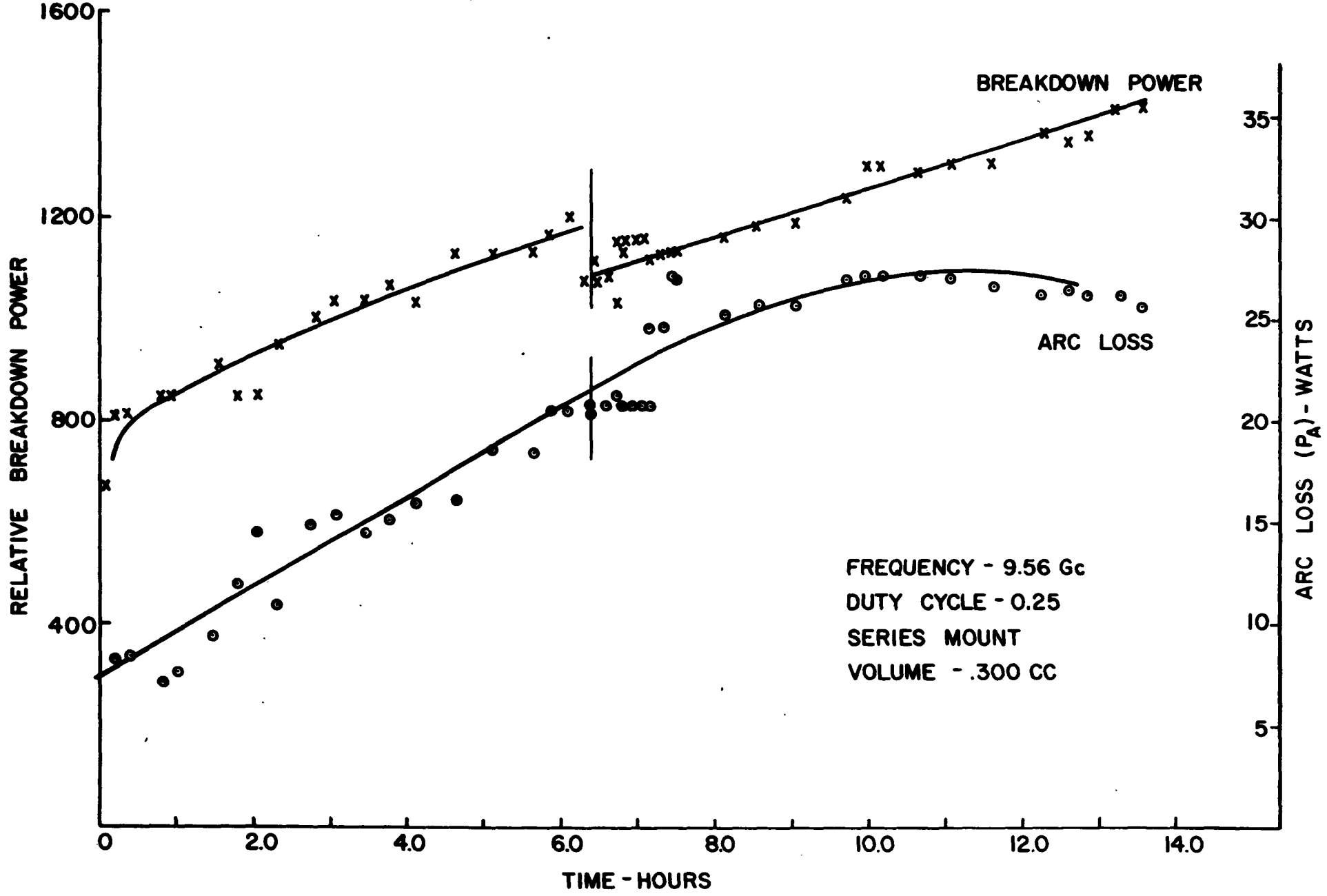
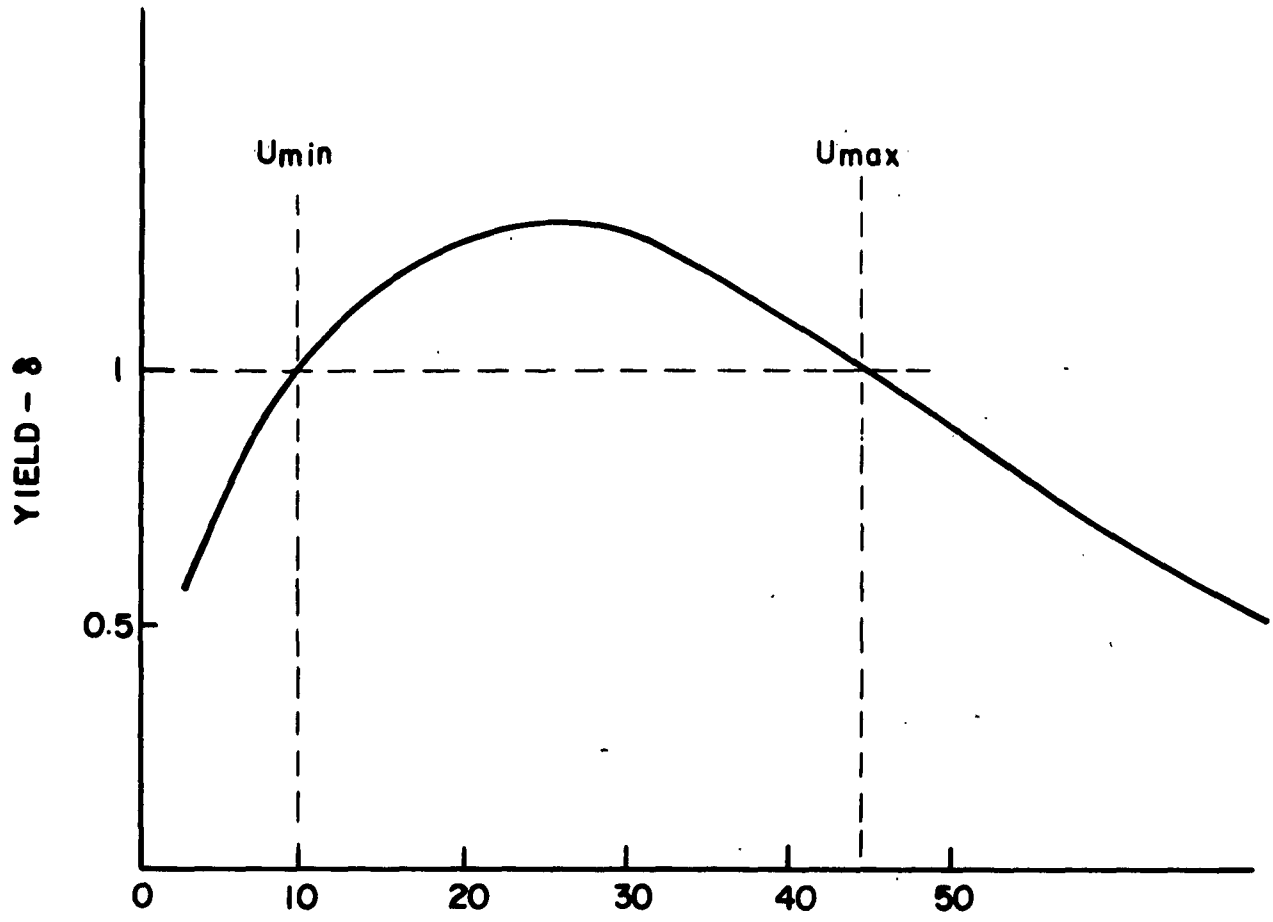


FIGURE 9
YIELD OF ELECTRONS DUE TO SURFACE BOMBARDMENT BY IONS



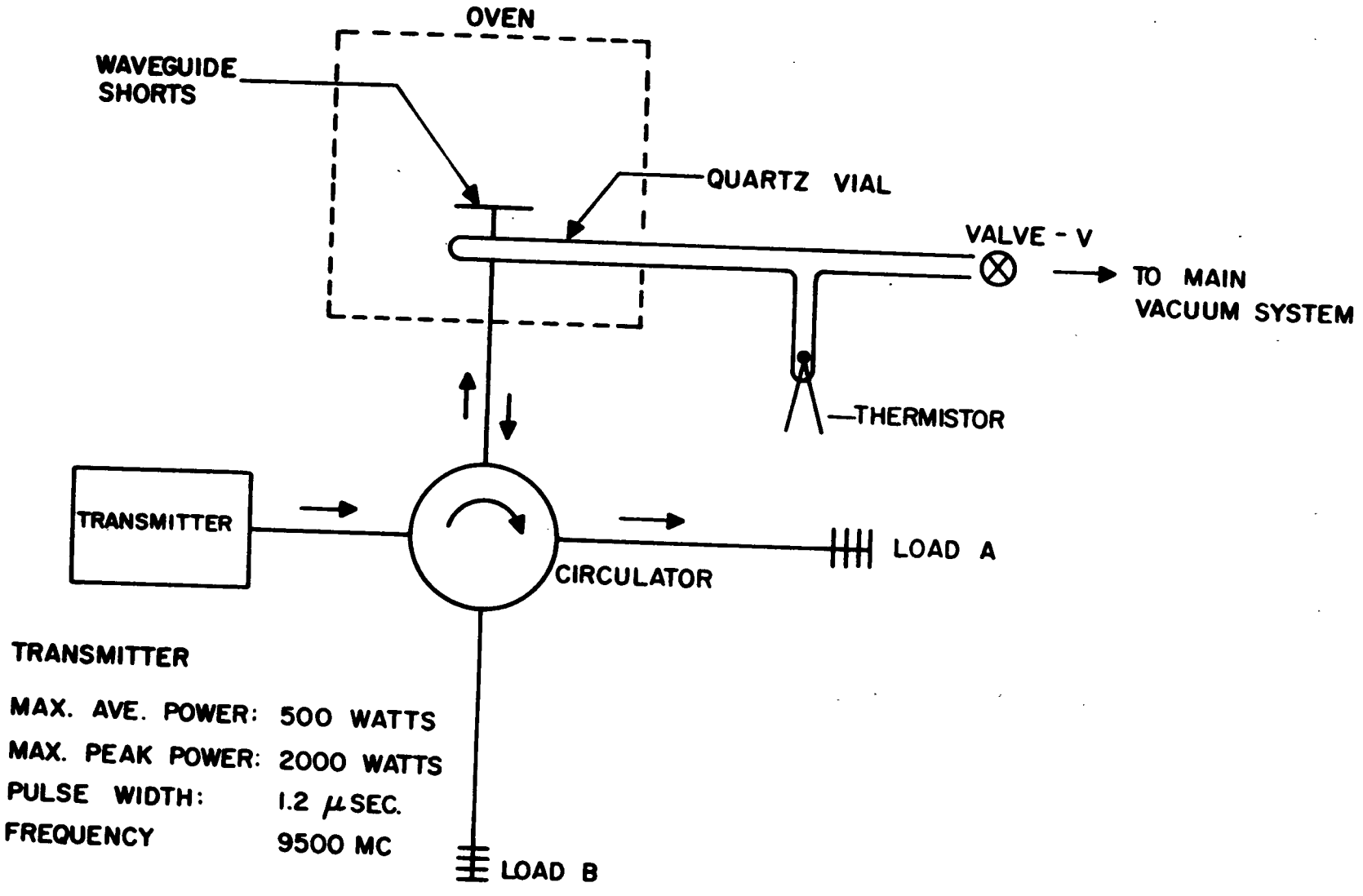


FIGURE 10

WAVEGUIDE AND VACUUM SYSTEM

FIGURE 11

QUARTZ VIAL OUTGASSING

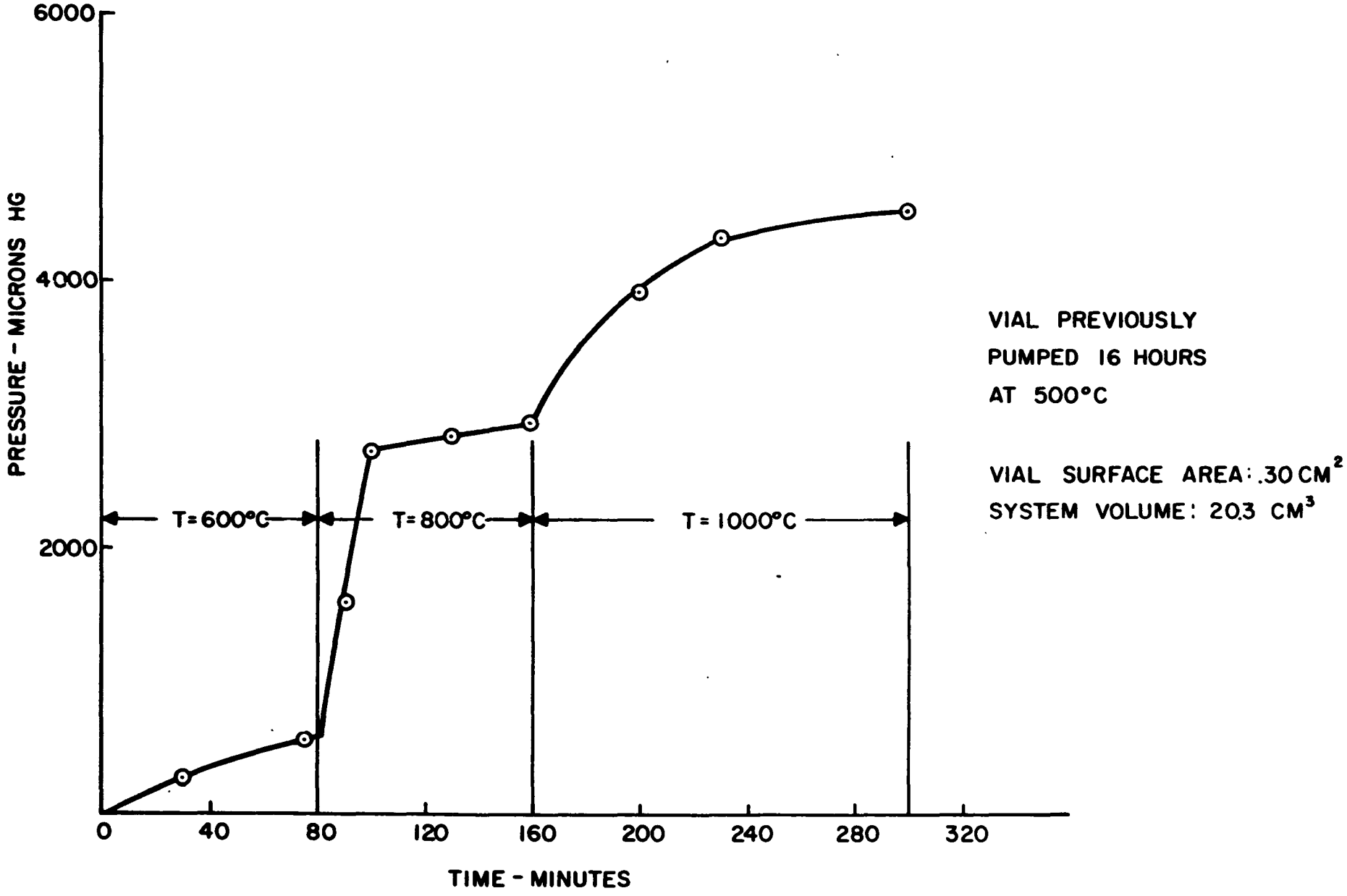


FIGURE 12

ARGON CLEAN UP-RECOVERY AT 200°C

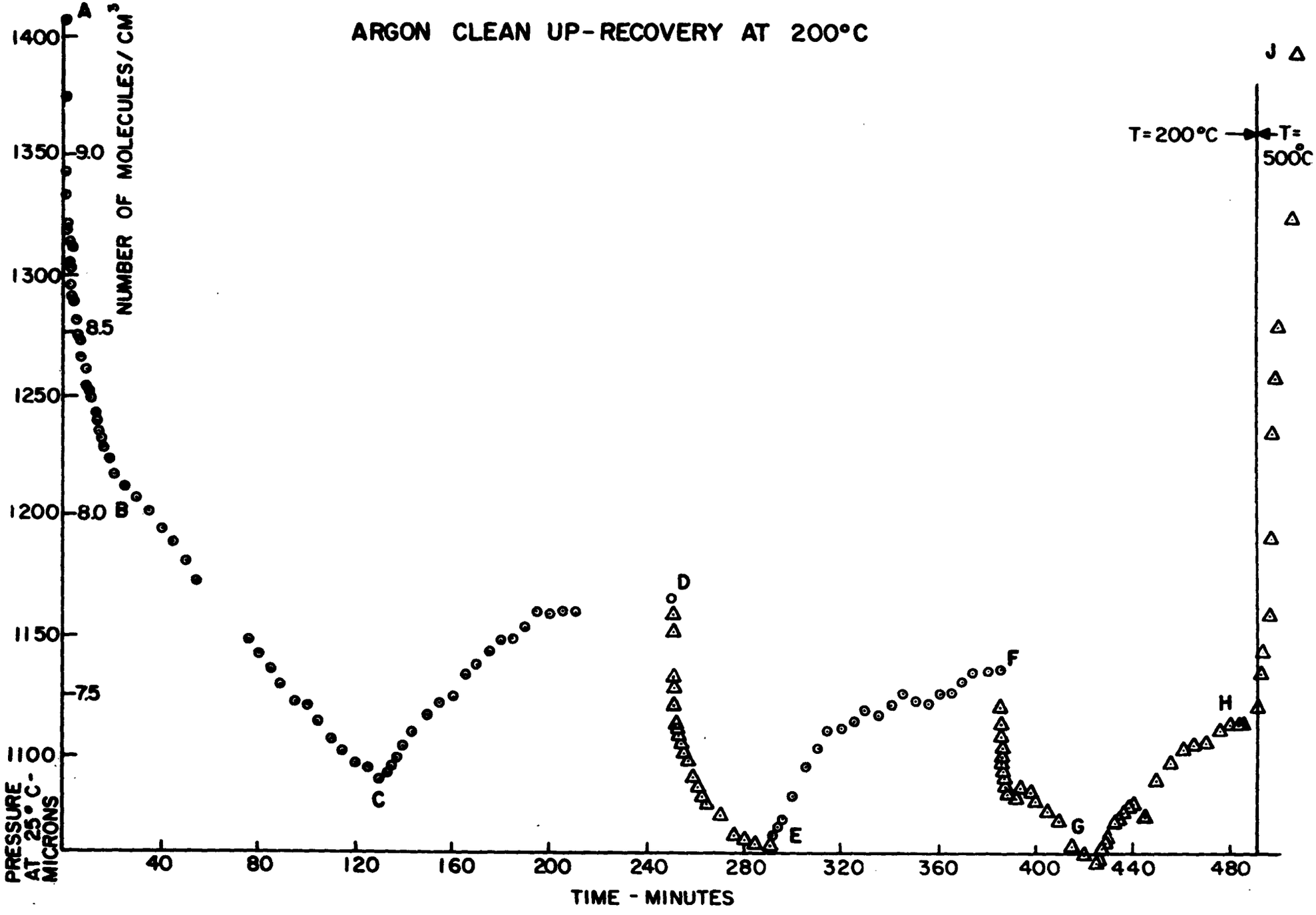


FIGURE 13

ARGON CLEAN UP-RECOVERY CYCLE AT 400°C

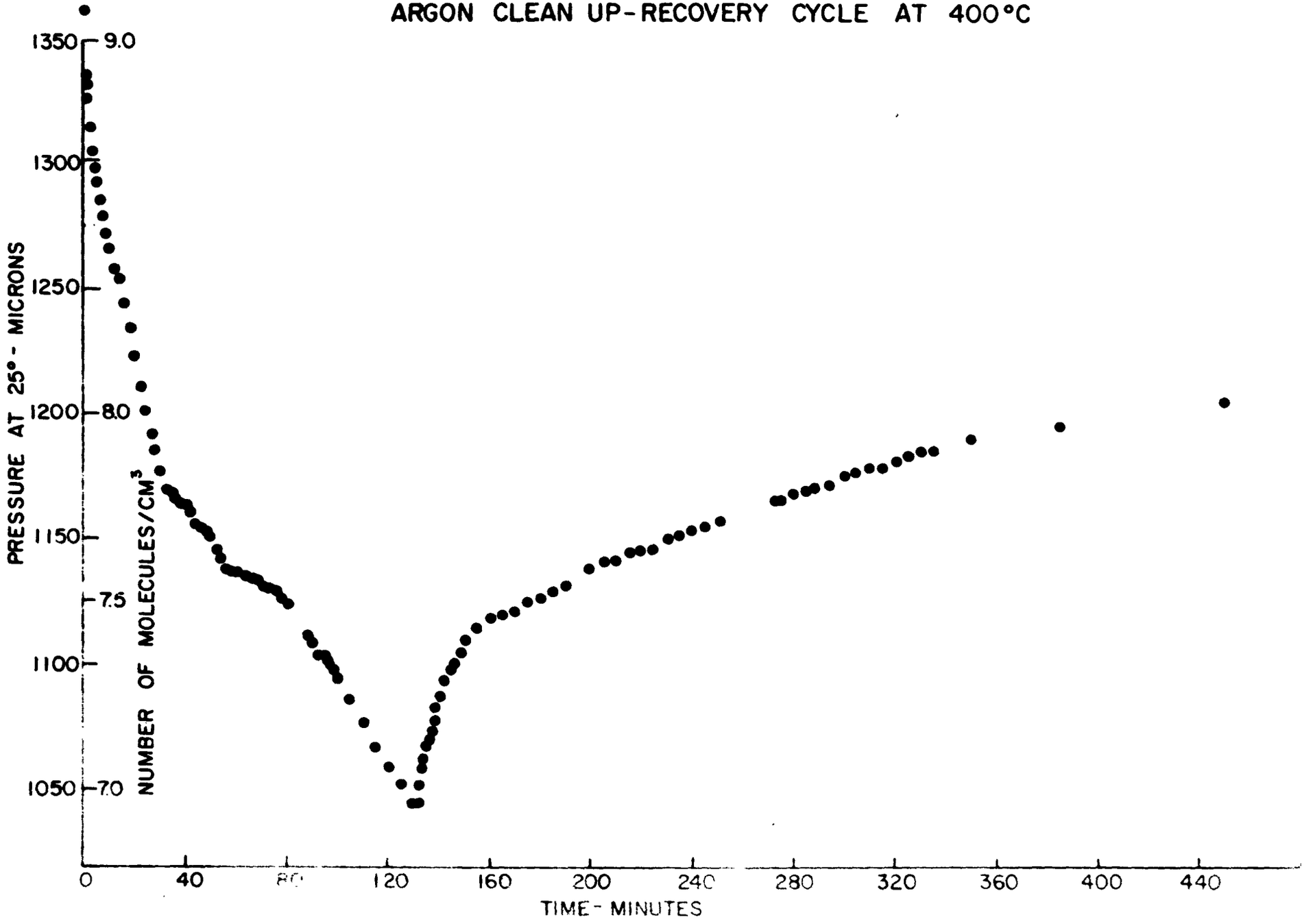


FIGURE 14

ARGON CLEAN UP-RECOVERY CYCLE AT 400°C

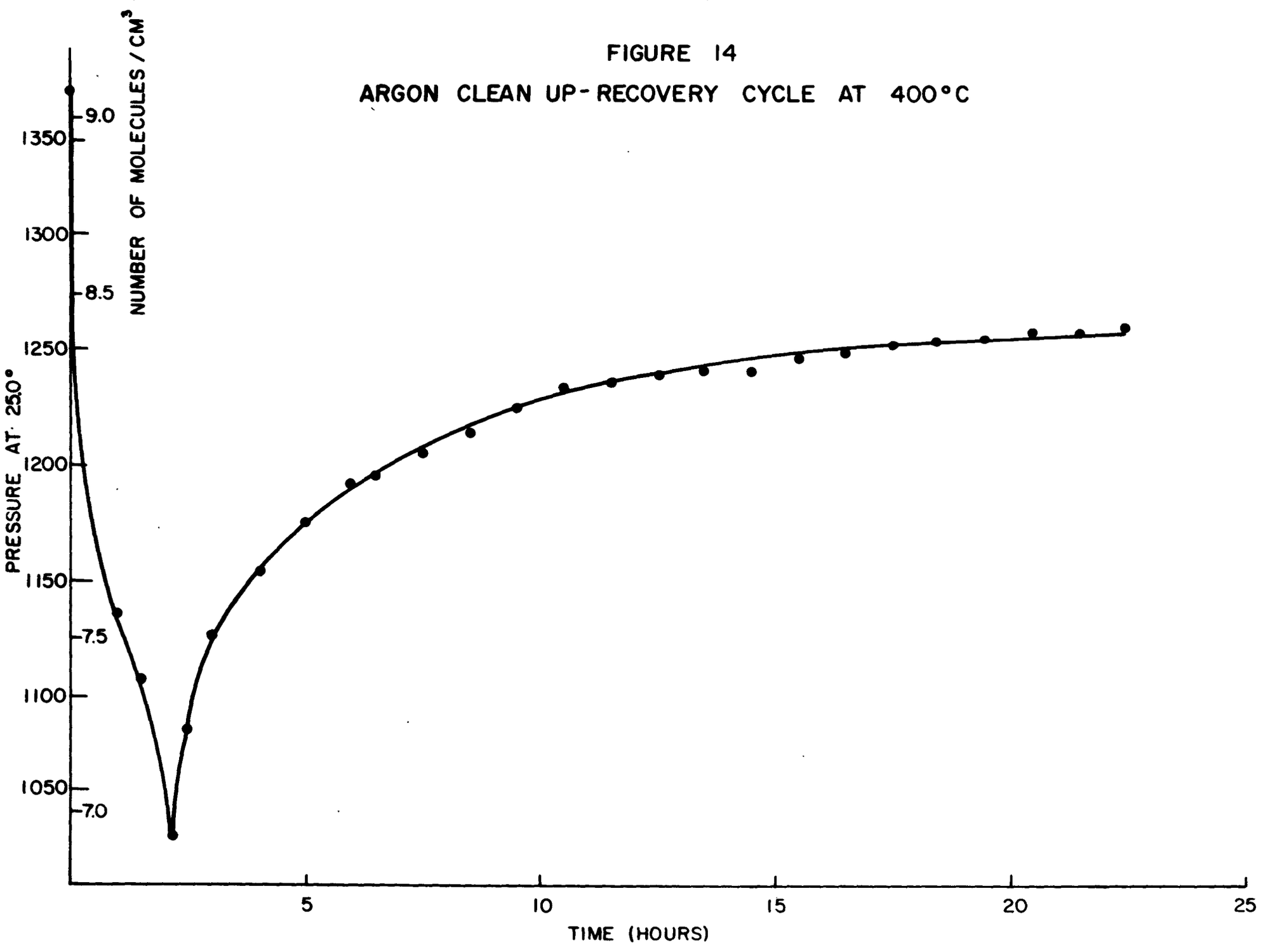


FIGURE 15

SPONTANEOUS EMISSION OF CLEANED UP ARGON ATOMS

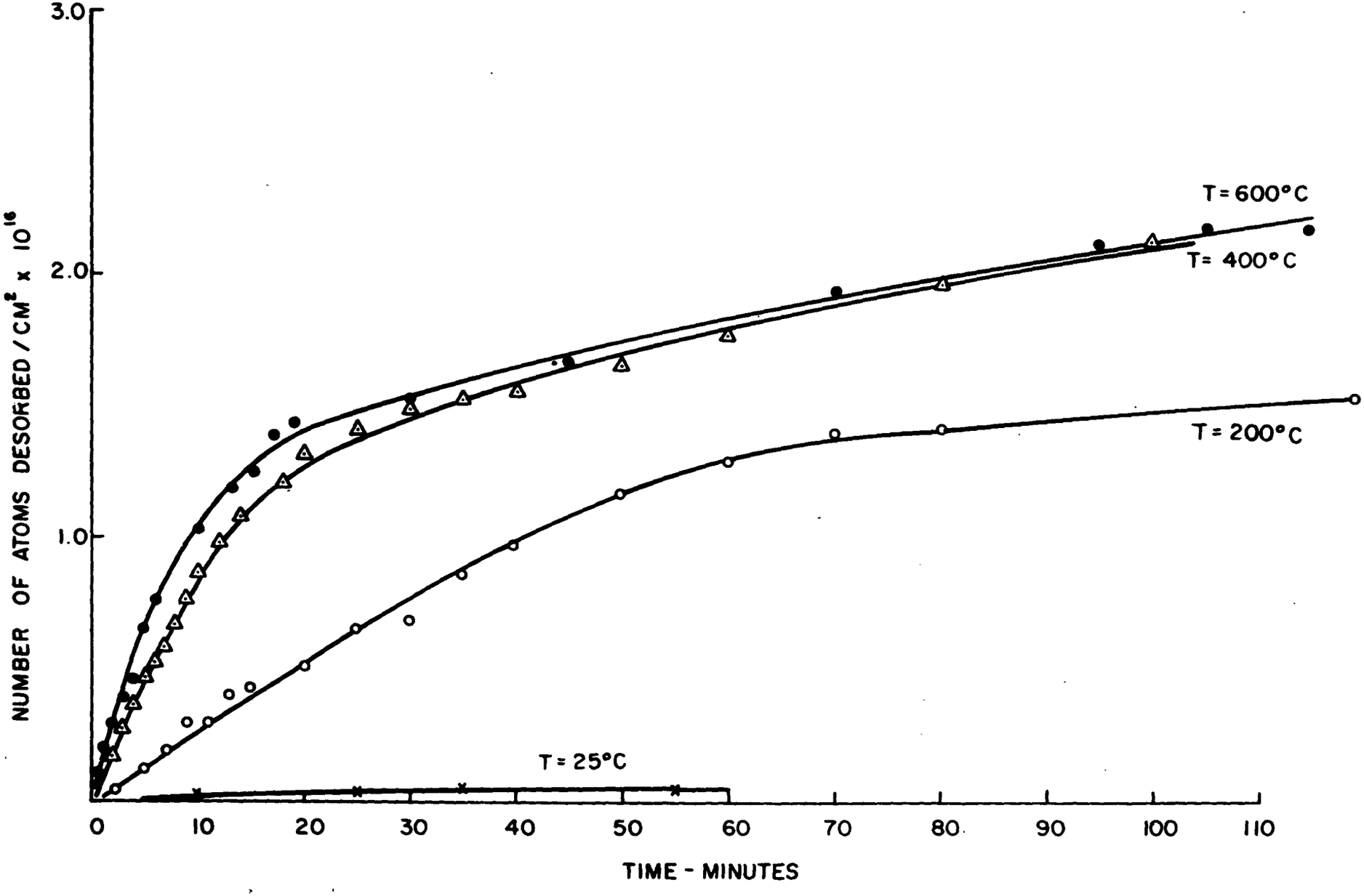


FIGURE 16

CLEAN UP OF ARGON ATOMS

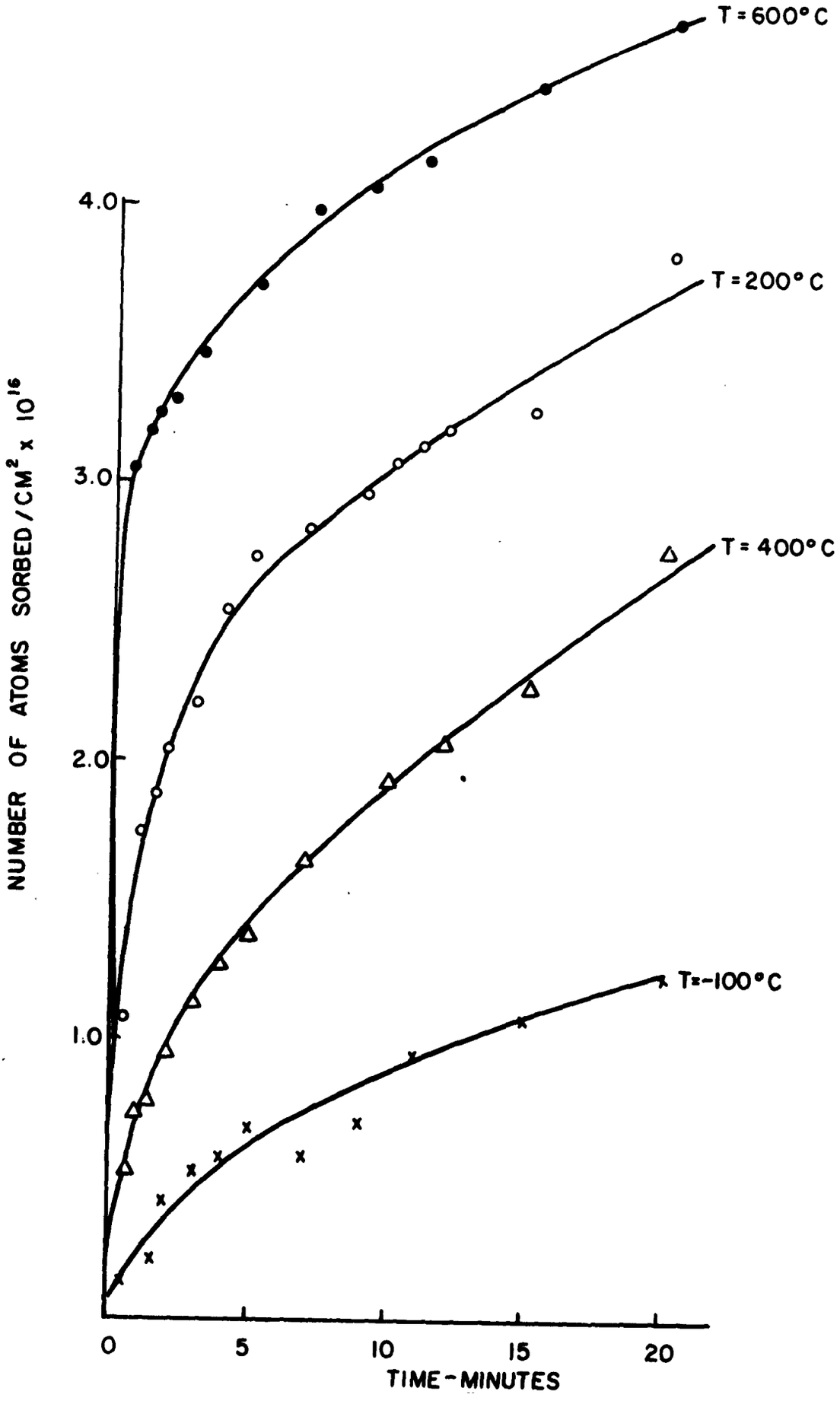


FIGURE 17

CLEAN UP OF ARGON ATOMS

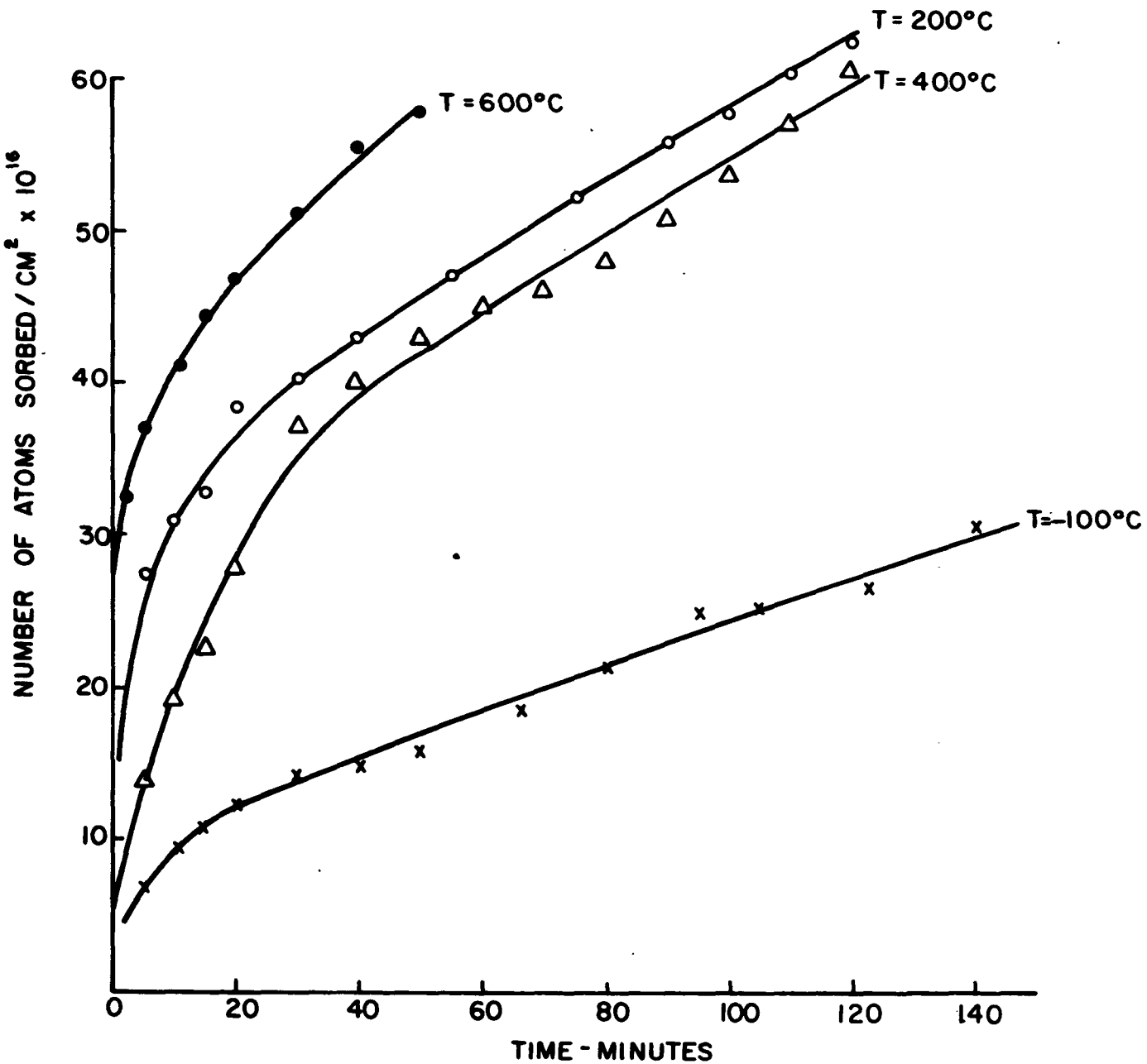
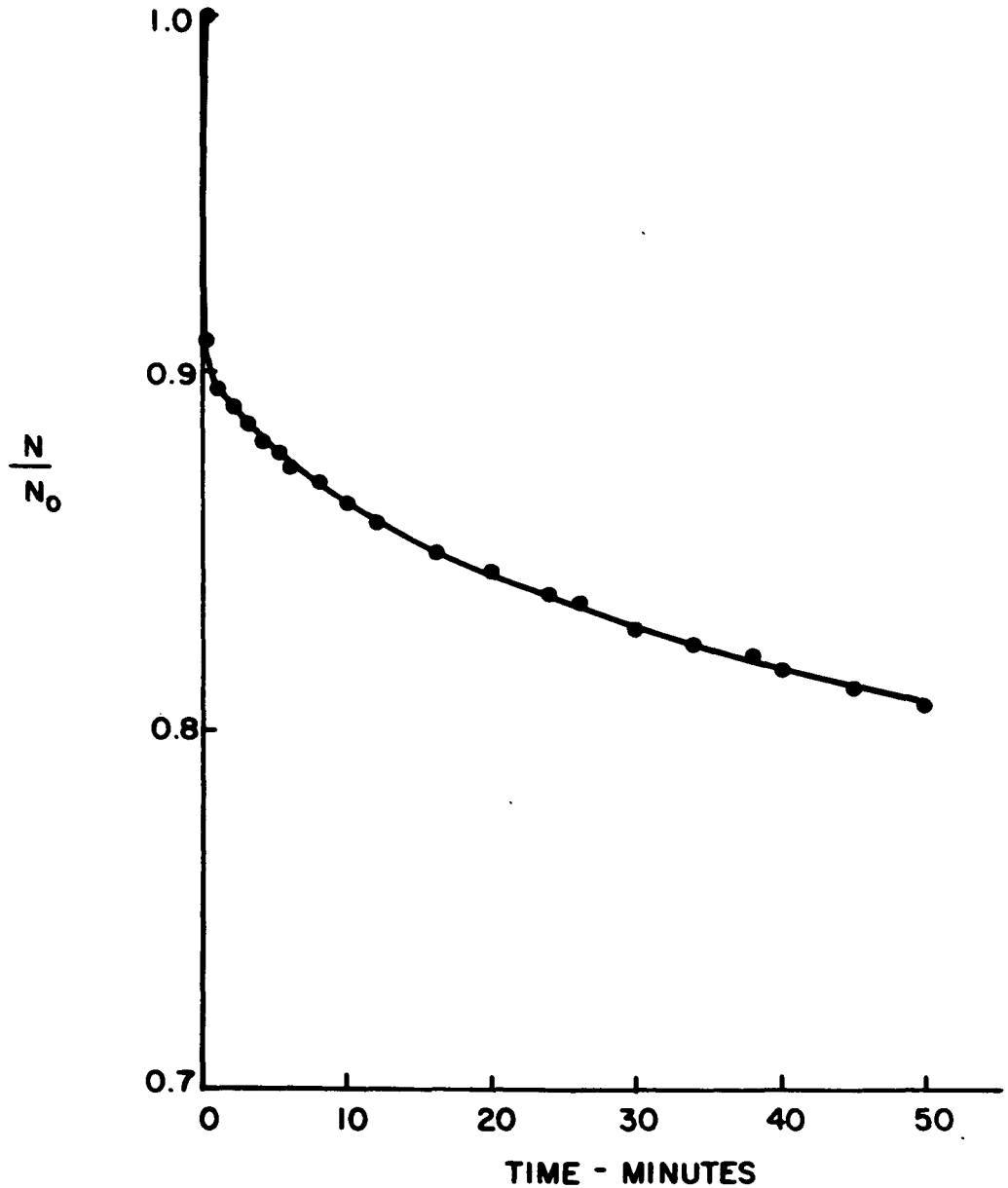


FIGURE 18

CLEAN UP OF ARGON ATOMS AT 600°C



●●● EXPERIMENT

—— THEORY

$$N = N_0\left[1-\alpha_1\tau_1\left(1-e^{-\frac{t}{\tau}}\right)-\alpha_2\tau_2\left(1-e^{-\frac{t}{\tau_2}}\right)-\alpha_3 t\right]$$

τ_1 = 3 SEC.

τ_2 = 9 MIN.

α_1 = 2.0 SEC.$^{-1}$

α_2 = 0.0044 SEC.$^{-1}$

α_3 = 0.0010 SEC.$^{-1}$

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INVESTIGATION OF HIGH POWER GASEOUS ELECTRONICS

Harold S. Maddix and Charles S. Ward

Final Progress Report January 1, 1962 to November 15,1962

58 pp. - Illus. - Graphs, Contract No. DA-36-039-SC-89161

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Final Progress Report
Contract No. DA 36-039-SC-89161

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